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Development of a Portable Monitor for Detection of Toxic Organic Compounds

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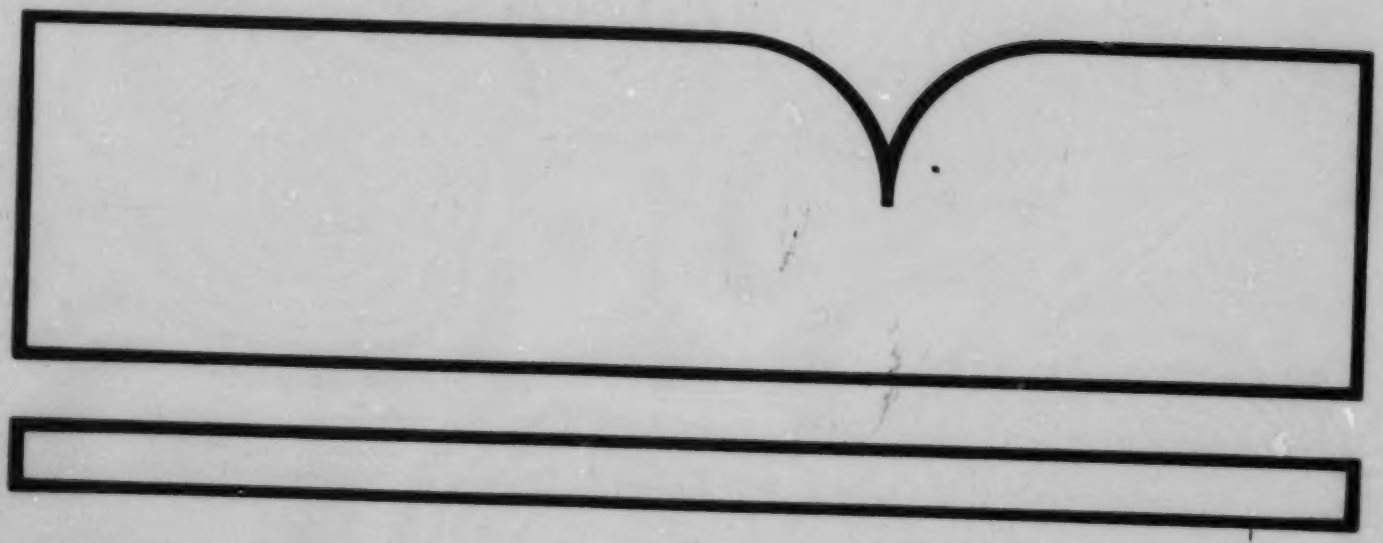

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DEVELOPMENT OF A PORTABLE MONITOR FOR DETECTION OF TOXIC ORGANIC COMPOUNDS

by

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16. ABSTRACT

A dual beam prototype instrument was designed, constructed, tested, and delivered to the Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, in December 1983. It was designed for monitoring benzene with the Hg 253.7 nm line using the TALMS technique. The instrument consisted of three units: the optical unit (weight: 52 lbs), the electronics unit (weight: 16 lbs), and a power supply (weight: 14 lbs). Tests of the performance of the benzene monitor showed linear response from the detection limit of approximately 40 to 3000 ppm-v. The magnetically contained lamps used with the TALMS technique were improved so that they are more stable, intense and have longer service lives. Searches for locations of potential TALMS signals in o-, m-, p-xylenes, bromobenzene, benzene and aniline were carried out. TALMS signals were found for p-xylene with Si, Co, and Sb lines at 252.9 nm. No signals were found at this wavelength for o- or m-xylenes. TALMS signals were also found for bromobenzene at 270.2 nm (Pt) and 266.5 nm (Fe) and aniline at 293.8 nm (Bi). It is recommended that further searches be carried out to extend the potential of the TALMS technique to other organic compounds and to decrease its detection limit for benzene. The use of a new related technique, Atomic Line Molecular Spectroscopy (ALMS), is also recommended.

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems for air, implementation of agency-wide quality assurance programs for air pollution measurement systems, and supplying technical support to other groups in the Agency including the Office of Air and Radiation, the Office of Toxic Substances, and the Office of Solid Waste.

Analysis of organic compounds in the atmosphere is a difficult task requiring a variety of analytical techniques to ensure that identification and quantification are valid. The environmental analyst requires methods that are precise, accurate and easy to implement. This study is concerned with the development of a new instrument which utilizes the information available in the high resolution absorption spectra of organic compounds in the ultraviolet and visible regions. The use of this instrument in the analysis of organic compounds may greatly improve the ability of the analyst to produce valid results.

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ABSTRACT

The objectives of this study were: a) to design, construct, and deliver a prototype portable tunable atomic line molecular spectrometer (TALMS) benzene monitor and b) to locate matches of atomic lines and sharp molecular absorption features in other toxic organic compounds. TALMS is a newly developed, high resolution molecular absorption technique that is used in the ultraviolet-visible region of the optical spectrum to detect molecules in the gas phase. The dual beam prototype instrument was designed, constructed, tested and delivered to the Environmental Monitoring Systems Laboratory, U. S. EPA, Research Triangle Park, North Carolina, in December, 1983. It was designed for monitoring benzene with the Hg 253.7 nm line using the TALMS technique. The instrument consisted of three units: the optical unit (weight: 52 lbs, dimensions: 28x9x9"); the electronics unit (weight: 16 lbs, dimensions: 19x12.5x5.25"); and a power supply (weight: 14 lbs, dimensions: 12x8x8"). The total weight was 82 lbs, which is much less than that of the benzene monitor previously developed (144 lbs). Tests of the performance of the benzene monitor showed linear response from the detection limit of approximately 40 ppm-v to 3000 ppm-v. Limited laboratory tests of a cryogenic concentration system for the unit were carried out. Details of the construction of the prototype are given in this report.

Lamps for the volatile elements (As, Pb, Zn) that can be used with the TALMS technique are now available commercially from Heraeus, Hanau, West Germany. The magnetically contained lamp used with the TALMS technique has been improved so that it is more stable, intense and has a longer service life.

Searches for locations of potential TALMS signals in o-, m-, p-xylenes, bromobenzene, benzene and aniline were carried out using high and medium resolution absorption spectra as guides. TALMS signals were found for p-xylene with Si, Co, and Sb lines at 252.9 nm. No signals were found at this wavelength for o- or m-xylenes. TALMS signals were also found for bromobenzene at 270.2 nm (Pt) and 266.5 nm (Fe) and aniline at 293.8 nm (Bi). Numerous other lines were tried but did not yield TALMS signals. These searches are slow and tedious due to incomplete or inadequate high resolution spectra.

It is recommended that further searches be carried out to extend the potential of the TALMS technique to other organic compounds and to decrease its detection limit for benzene. The use of a related technique, Atomic Line Molecular Spectroscopy (ALMS), that does not use the Zeeman effect, should require lighter equipment and have much lower detection limits. However, the selectivity of ALMS would not be as good as TALMS.

This report was submitted in fulfillment of Interagency Agreement AD-89-F-3A403 by the Lawrence Berkeley Laboratory under the sponsorship of the U. S. Environmental Protection Agency. This report covers the period from October, 1982, to November, 1983, and the work was completed as of November, 1983.

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SECTION 1

INTRODUCTION

Tunable atomic line molecular spectroscopy (TALMS) is a new high resolution, molecular absorption approach to monitoring organic vapors that is different from most present analytical techniques [1,2]. Whereas present methods depend on some form of chromatographic separation for compound identification, the TALMS technique depends only on ultraviolet-visible absorption properties. There is no separation procedure involved. It is highly specific because it responds only to very sharp rotational-vibrational molecular absorption features. Previous studies of TALMS [3,4] have resulted in construction and evaluation of prototype instruments for general laboratory use and for monitoring benzene. The goals of the present study are to: a) design, construct, and deliver to the Environmental Monitoring Systems Laboratory, U. S. EPA, a portable TALMS benzene monitor and b) to determine the spectral location of TALMS features in toxic organic compounds other than benzene. A portable TALMS instrument would be useful in field detection and monitoring for benzene and other toxic compounds. Determination of spectral locations of TALMS signals is necessary to optimize instrument performance and to extend the technique to other organic compounds.

The instrument design, construction, and testing are described in Section 3. The procedures and results of TALMS molecular absorption-atomic line matches are described in Section 4. Developments in lamp design and construction are given in Section 5. Appendices A and B include operating instructions for the TALMS portable monitor and detailed descriptions of the optical, mechanical, and electronic components.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Design and construction of a prototype transportable TALMS instrument for benzene detection was completed on schedule and delivered to the Environmental Monitoring Systems Laboratory, Environmental Protection Agency at Research Triangle Park, North Carolina in December, 1983. It consisted of three modules: a sensing module, an electronics module and a power supply. The sensing module weighs 52 lbs (24 kg) and the electronics module and power supply 16 lbs (7.3 kg) each. The electronics module may be removed from the others, connected only by an extension cord, if this makes field use more convenient. The electronics module was designed to be powered by a motor generator (100 watts of power are required) that could be removed from the sampling site. Instrument performance tests show that the instrument has a linear response over the concentration range from 40 to 3000 ppmv and a lower limit of detection of about 40 ppmv. This detection limit is too high for ambient air detection of benzene and some form of concentration technique will be needed for this purpose.

A new commercially available light source (W. C. Heraeus GmbH, PEW, Hanau, West Germany) is available that allows As, Pb, and Zn lines to be used in this instrument in addition to the Hg lines for which it was designed. However, different interference filters or a monochromator would be required if these lamps were used. While this instrument was under construction, major advances were made in the assembly and operation of the magnetically confined atomic discharge lamps leading to more stable and intense emission.

Searches for TALMS signals in organic molecules were found to be slow and tedious due to lack of high resolution absorption spectra in the literature for most compounds. After considerable experimentation with a variety of lamps and medium resolution absorption spectra, TALMS signals were found for p-xylene (Si: 252.85 nm; Co: 252.9 nm; Sb: 252.85 nm; Pt: 273.4 nm ; Pt: 248.7 nm); bromobenzene (Pt: 270.2 nm; Fe: 266.5 nm); aniline (Bi: 293.8 nm); and benzene (Si: 252.85 nm; Sb: 252.85 nm). No signals were found for o- or m-xylene. TALMS signals have now been found for benzene, bromobenzene, chlorobenzene, toluene, p-xylene, aniline, phenol, pyridine, and formaldehyde [1-4].

Since major decreases in detection limits are dependent upon the location of more intense analysis lines, it is recommended that more studies be carried out to locate new analysis lines for compounds of interest.

It is also recommended that the investigation of a related analytical technique called Atomic Line Molecular Spectroscopy (ALMS) be initiated. This technique involves absorption near the intense absorption bands of the vacuum ultraviolet (180.0 to 220.0 nm) region using one or more atomic lines. The advantages over the TALMS technique are much lower detection limits and a lighter instrument since only a small magnet and no squeezer are required. Selectivity will be decreased, but it may be adequate if absorption by several atomic lines is utilized.

SECTION 3

INSTRUMENT DESIGN AND CONSTRUCTION

There is a great need for portable or transportable instruments to detect and monitor specific organic pollutants in ambient air and near sources such as waste disposal and industrial production sites. Direct monitors are needed for specific chlorinated hydrocarbons and various aromatic hydrocarbons including benzene, toluene and other substituted benzenes. Since substituted benzenes are known to produce TALMS signals, or have the potential for doing so, a transportable TALMS instrument was designed for their determination. This unit was specifically designed for benzene determinations but can be later modified for the determination of other compounds.

The approach was to construct a light weight unit by separating the sensing elements of the TALMS system (light source, magnet, cell, and photomultiplier tube) from the power supply and electronics. Direct monitoring could then be achieved, if pollutant concentrations are high enough, by carrying the sensing unit into the region to be monitored while it remains connected by a cord to the power source. If concentrations are too low for direct detection by the sensing unit, an appropriate cryogenic trapping unit could be used to concentrate the samples before injection into the field unit. The unit constructed was designed for benzene detection and utilized the Hg 253.7 nm line in a double beam configuration. It was based upon a previous design for a benzene monitor [4].

The main limitations in decreasing the size and weight of the TALMS instrument are the necessity for the permanent magnet and squeezer components with the instrument. Both of these components are quite heavy. As a result, it is unlikely that any further major reduction in size or weight will be achieved. Consideration was given to a smaller permanent magnet system. However, commercially available, stable Hg lamps could not be used with this smaller system. Therefore, the present magnet was used.

The prototype instrument that resulted had a total weight of 82 lbs (37 kg) as compared to a weight of 144 lbs (65 kg) for the benzene monitor developed previously [4]. The optical unit weighed 52 lbs (24 kg) and had dimensions of 28" x 9" x 9" (71 x 23 x 23 cm). The electronic unit weighed 16 lbs (7.0 kg) and had dimensions of 19" x 12.5" x 5.25" (48 x 32 x 13 cm). The power supply weighed 14 lbs (6.4 kg) and had dimensions of 12" x 8" x 8" (30 x 20 x 20 cm). Further details of the design and construction of the instrument are given below. The operating instructions, details of components, and circuit diagrams are given in Appendices A and B. The instrument that resulted from following this

approach is well suited for, or can readily be adapted to, most field monitoring needs.

Optical Components

The optical components are mounted on a light weight, aluminum optical bench. Figure 1 is a diagram of the optical arrangement, and Figures 2 and 3 are photographs of the instrument. The optical components consist of a small permanent magnet (Permag Control Corporation, Elk Grove Village, IL), a squeezer (Lawrence Berkeley Laboratory), a 6.0 inch (15 cm) sample cell constructed of vacuum tight, mini-flange fittings (Varian Associates, Palo Alto, CA), and photomultiplier tubes with a 1P28 spectral response (Hamamatsu Corporation, Middlesex, NJ). The light source is a low pressure mercury lamp (Oriel Corporation, Stamford, CT) of small diameter (6.5 mm O.D.) that fits into the small magnet pole gap. An interference filter (Oriel corporation, Stamford, CT), mounted at the entrance to the beam splitter is used to isolate the mercury 253.7 nm line. If other spectral regions must be isolated to allow a greater number of compounds to be monitored, the small, light weight J-Y, model H-10 monochromator (J-Y Optical Systems Division, Instruments S.A. Inc., Middlesex, NJ) can be installed to replace the interference filter.

Electronic Components

The electronic components are contained in a small, aluminum electronic bin (Europac System, Schroff, W-Germany). The Eurorack contains a lock-in-amplifier, (from Grun KG, Wetzlar, W-Germany), a squeezer driver and lamp driver, a two channel automatic gain control, and signal processors for the two photomultiplier tubes. The last named units were fabricated at the Lawrence Berkeley Laboratory. It also contains a two channel log amplifier (Analog Devices 757N) and two power amplifiers produced by the SanKen Co. (SI-105-0G); one for the squeezer and one for the lamp. This module is attached to the optical components module with an extension cord and need not be near the optical unit. Schematics of the circuits in these units are contained in Appendix B. Only about 100 watts of power are required to operate the instrument which can be easily supplied by a small motor generator. At least five corporations manufacture such motor generators (e.g. Pioneer Gen-E-Motor Corp., Chicago, IL). The units typically weigh about 70 lbs (32 kg). A very long extension cord can be used to connect the instrument modules to the motor generator if danger of contamination exists.

Instrument Performance

Analytical curves covering the range from 0 to 820 ppmv and 0 to 4000 ppmv are presented in Figures 4 and 5. They were obtained by withdrawing a known volume of vapor in equilibrium with solid benzene at 0 ^{o}C and injecting it into the absorption cell of the instrument. Knowing the partial pressure of benzene in equilibrium with the solid and the

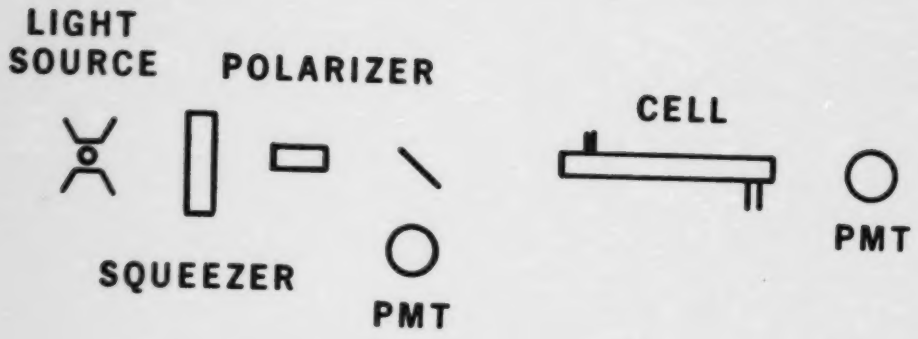

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Figure 1. Optical components of the transportable TALMS instrument.

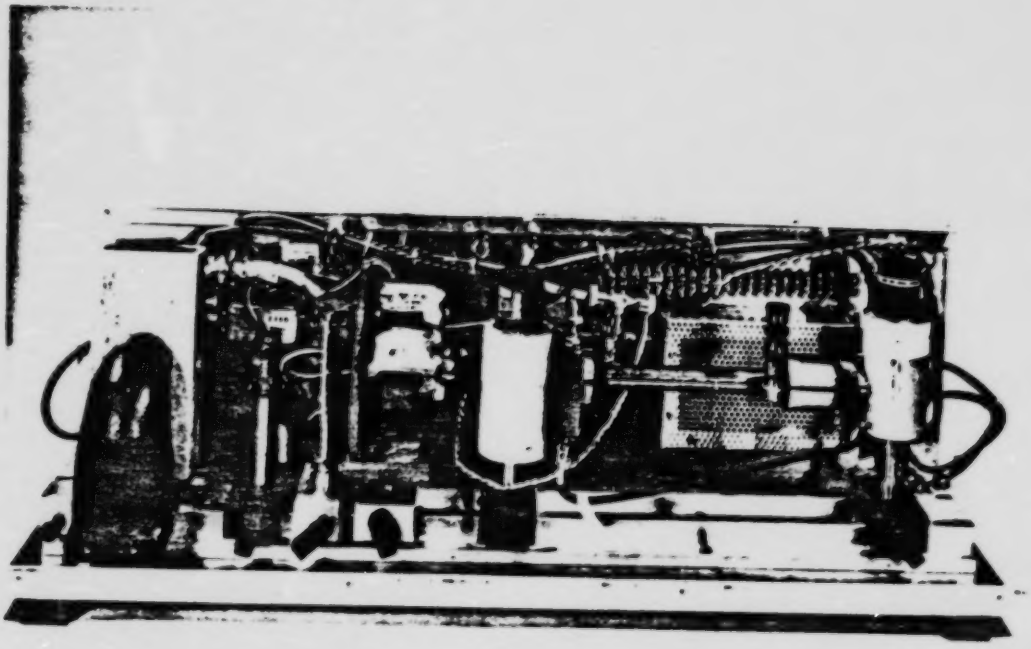

Figure 2. Photograph of TALMS sensing module.

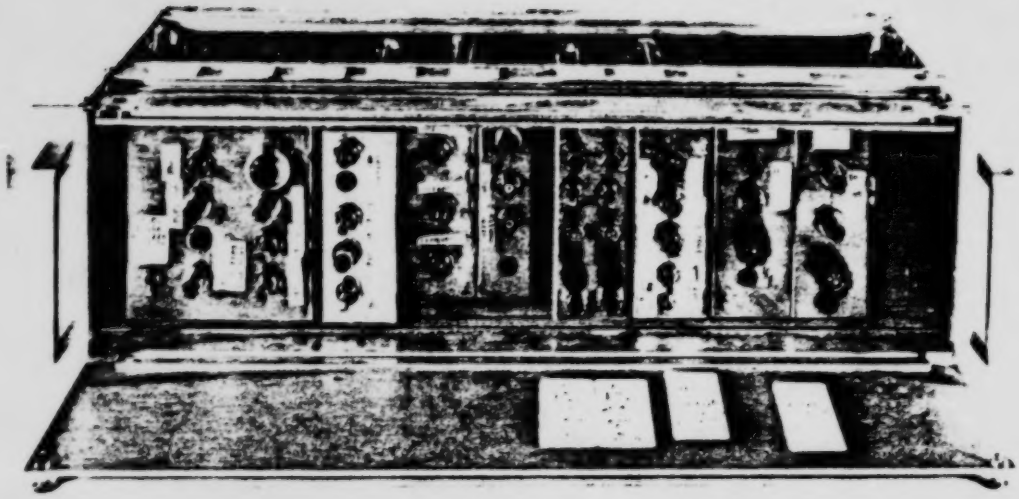

Figure 3. Photograph of TALMS electronic module.

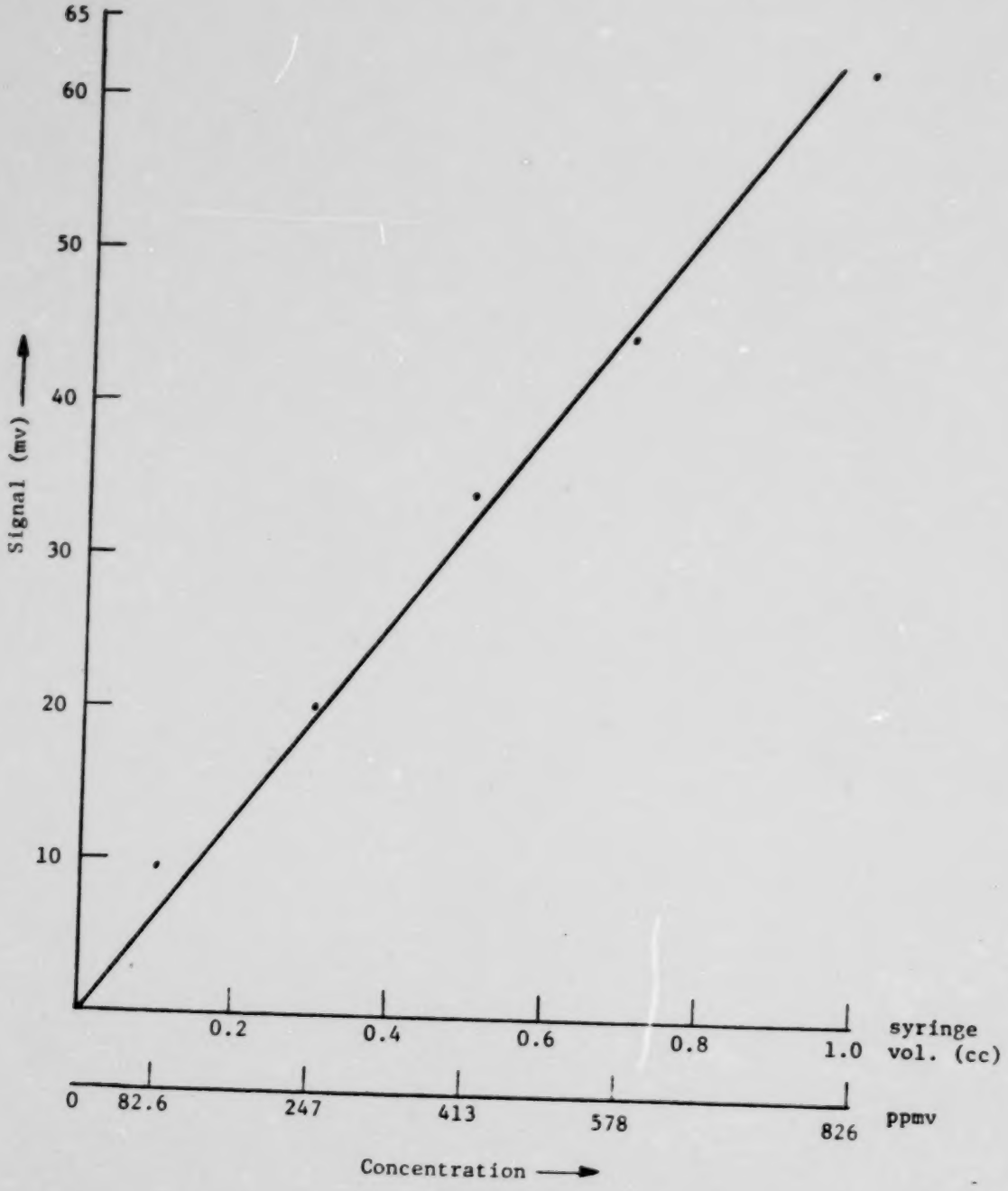

Figure 4. Low concentration calibration curve for benzene.

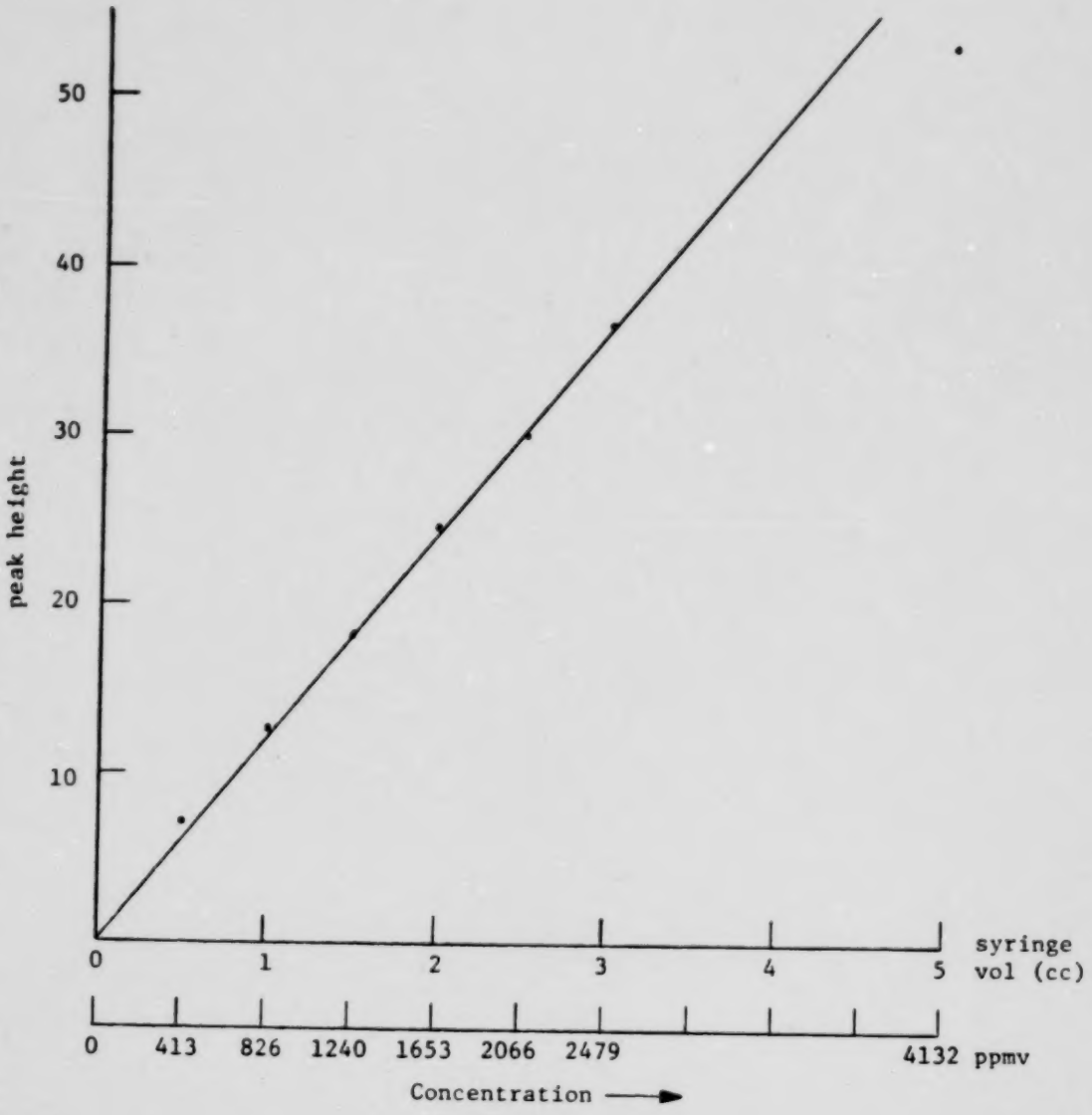

Figure 5. High concentration calibration curve for benzene.

volume of the cell, the concentration of the benzene in the cell (ppmv) could be calculated. The volume of the vapor transferred and the corresponding concentrations are indicated in Figures 4 and 5. The curves are linear over the range tested.

To determine an approximate lower limit of detection, the noise level was measured while data for the analytical curves was being collected. During the low concentration measurements baseline noise was 4-5 mv corresponding to 22 ppmv. Using a signal to noise ratio of two, the lower limit of detection would be 44 ppmv.

Cryogenic Trapping

Since the detection limit of the TALMS instrument is too high for direct ambient air monitoring of benzene, preliminary laboratory tests of a concentration system were made. The cryogenic system is sketched in Figure 6. This system is designed to condense materials from an air stream that have low vapor pressures at the boiling point temperature of liquid nitrogen. The procedure is to cool the trap by immersion in liquid nitrogen, move the desired quantity of air through the trap, allow the liquid oxygen to boil off, evacuate, heat to ca. room temperature, and finally allow incoming air to push the trapped material into the TALMS absorption cell. A severe temperature gradient exists in the trap in this system while organic compounds are being concentrated from the flowing gas stream. The heating coil keeps the upper part warm to prevent water vapor from freezing and plugging the inlet while the lower part is cold because of immersion in liquid nitrogen. Initially oxygen condenses in the lower portion of the trap, but as the level rises, it reaches a point where it is being boiled off as fast as it condenses. Hence, the amount of air that can be pulled through the trap is not limited by oxygen condensation. Early experiments demonstrated as much as 400 L of air could be pulled through the trap without plugging. Of course, this involved rather long sampling times (ca. 40 min). For the present test only ca. 5 L of air were pulled through in order to shorten the time required for testing.

Before use the trap must be "blanked" by heating with air flowing through until no TALMS signal is observed. After immersion in liquid nitrogen, it takes the trap about four minutes to cool to a point where benzene does not pass through. This was determined by injecting benzene vapor through the septum (see Figure 6) with air flow through the system and noting the appearance of a TALMS signal. After the trap had cooled, a blank was run by pulling room air through the trap for 5 minutes at a rate of 4.5 L/min. No signal was observed upon warming up the trap. Next, benzene was slowly injected into the air stream during a 5.5 minute interval while air was being pulled through at a rate of 1 L/min. This procedure was repeated twice using 5 mL of benzene vapor to demonstrate the reproducibility of the system and once by direct injection into the cell to demonstrate complete recovery.

Because of the presence of the liquid oxygen, some care must be exercised after the trapping step. If the temperature is allowed to increase too rapidly, sufficient pressure will build up in the trap to

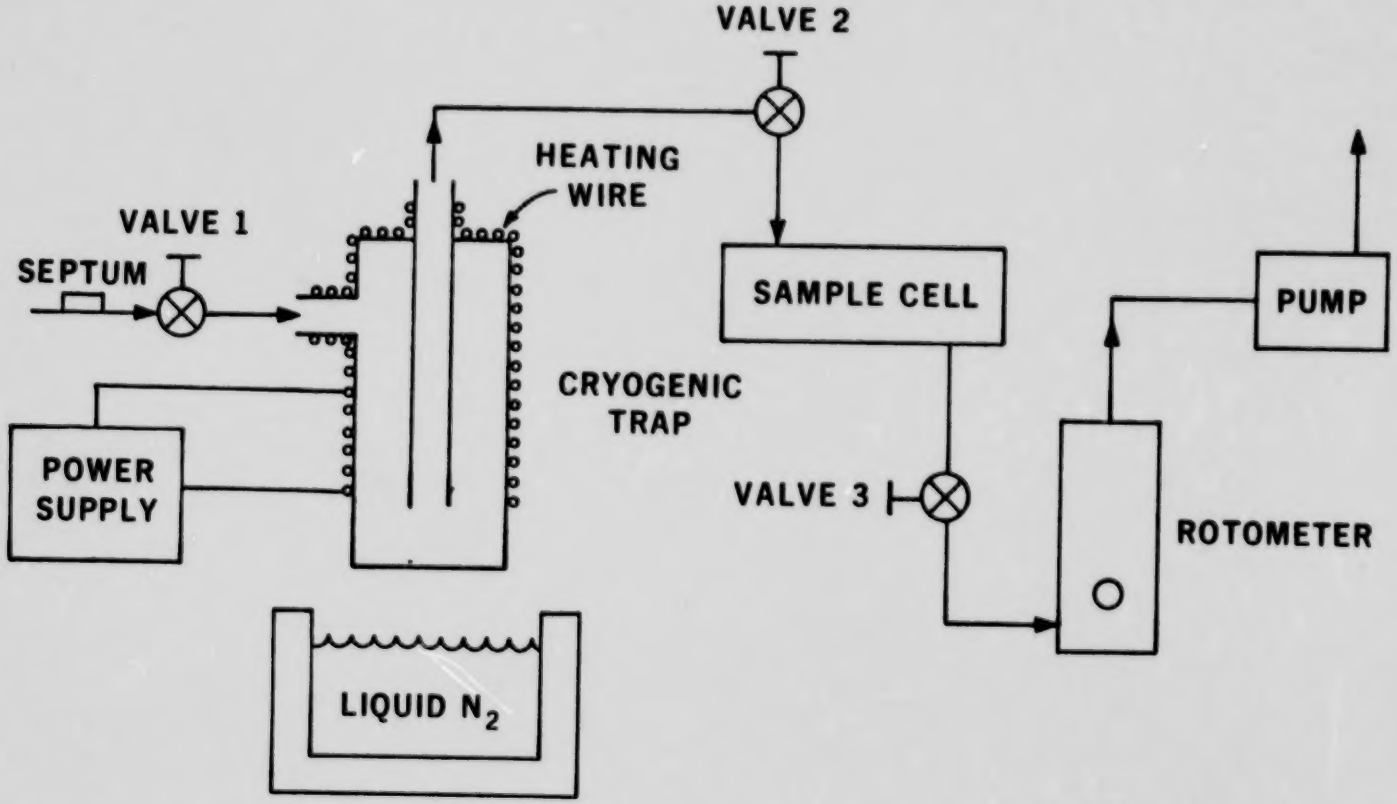

Figure 6. Cryogenic trapping system.

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cause the connections to be blown off. This can be avoided by disconnecting the pump and allowing the oxygen to escape freely. The flow may be routed through the sample cell to determine if any benzene is escaping. If the flow becomes too rapid, the liquid nitrogen dewar can be raised to retard the boiling of liquid oxygen. Of course the problems with liquid oxygen can be eliminated by using liquid argon as the cryogenic fluid if it is available.

SECTION 4

MOLECULAR ABSORPTION - ATOMIC LINE MATCHES

The TALMS technique is capable of detecting a very large number of different organic molecules. A molecular absorption feature in the ultraviolet - visible region that shows a change of a few tenths of an absorbance unit over a wavelength interval of about 0.01 nm is required. An atomic emission line must also occur at about the same wavelength. Literature searches indicate that many organic molecules possess sharp absorption features. However, data in the literature are incomplete for most compounds, nonexistant for others, and sometimes inaccurate. Atomic line compilations show that lines can be found in any wavelength region although these lines are often very weak. Finding matches is a tedious process because in many literature citations molecular absorption is not accurately measured, and because quiet, intense light sources that operate in a magnet field are not easy to produce. However, advances in the application of TALMS to multicomponent analysis at low concentration depend on the determination of more spectral regions of potential TALMS signals.

Recent improvements in the construction of light sources has greatly improved the probability of finding matches (see Section 5). The general method of finding matches for potential TALMS signals involves the following steps:

a) Locate crude spectral features, if available, in the literature,

b) Measure sharp absorption features using a medium resolution spectrograph,

c) Match the wavelength of these features to appropriate atomic lines found in the literature,

d) Construct light sources to produce the chosen atomic lines,

e) Search for a TALMS signal at the wavelength of the molecular feature with large amounts of the compound in the instrument,

f) Determine the purity of the compound used, if a signal is detected.

The U. S. Environmental Protection Agency is interested in future monitoring of organic compounds, many of which could potentially be determined by the TALMS technique. These compounds include 13 substituted benzenes: benzene, benzyl chloride, chlorobenzene, cresols, nitrobenzene, phenol, toluene, xylenes, and p-dichlorobenzene. Since benzene and substituted benzenes are known to have sharp absorption features in the ultraviolet region, matches were sought that could lead to TALMS detection.

Line Matches For Xylene

Low resolution spectral measurements [5] indicated that o-xylene has reasonably sharp absorption features in the region between 250 and 275 nm. To locate sharp absorption features in o, m, and p-xylene, medium resolution spectra (ca. 100,000) were obtained in second order with a JACO 3.4 m spectrograph (reciprocal dispersion = 2.6 Å/mm). Densitometer tracings of the absorption spectra at 30 °C recorded on photographic plates are displayed in Figures 7, 8, and 9. From the locations of the sharp absorption features and the known locations of atomic emission lines, it was decided that the following elements might provide matching lines: Fe, W, Si, Sb, Co, Zn, Ge, Mo, Os, Au, and Pt. Lamps providing intense spectra were made with all these elements except W, Os, and Mo.

A search for a TALMS signal was made using o, m, and p-xylene with each of the lamps that produced a good emission spectrum. The approach was to put a small amount of liquid into the cell. Then, using an emission line that overlapped a region of strong absorption, the pressure was adjusted by evacuating until the light was ~ 50% absorbed. Finally, different emission lines were chosen by adjusting the monochromator, and a TALMS signal was sought using 6 and 14 kG magnetic fields. All searches were made using the parallel configuration of magnetic field and light emission. Generally, the parallel configuration results in larger TALMS signals than does the perpendicular configuration. Not all apparent matches chosen on the basis of medium resolution absorption spectra actually resulted in a TALMS signal. However once the equipment has been assembled, it is a simple matter to test additional atomic lines.

The following lines were tried using o-xylene - Zn (307.2, 280.0, 277.0, 275.6, 260.8, 213.8 nm); Ge (265.1, 280.0, 277.0, 275.4, 270.9, 269.1, 259.2 nm); Au (267.6, 242.8 nm); Pt (264.7, 265.1, 265.9, 270.2, 270.6, 271.9, 273.4, 262.8, 248.7 nm); and Fe (302.0, 271.9, 252.2, 249.6, 248.9, 248.8, 248.4 nm).

The following lines were tried using p-xylene - Si (288.1, 253.2, 252.8, 252.4, 251.9, 251.6, 251.4, 250.7 nm); Fe (299.4, 298.3, 297.3, 296.6, 272.0, 271.9, 249.0, 248.8, 248.3, 247.9, 247.2, 246.2 nm); Co (252.9 nm); Sb (252.9 nm); Zn (307.2, 280.0, 277.0, 275.6, 213.8 nm); Au (267.6, 242.8 nm); and Pt (264.7, 265.1, 265.9, 270.2, 270.6, 271.9, 273.4, 262.8 248.7 nm).

The following lines were tried using m-xylene - Zn (307.5, 307.2, 280.0, 277.0, 275.6, 258.2, 260.8, 213.8 nm); Ge (280.0, 277.0, 275.4, 270.9, 265.1, 260.7, 259.2, 258.2 nm); Au (267.6, 242.8 nm); and Pt (264.7, 265.1, 265.9, 270.2, 270.6, 271.9, 273.4, 262.8, 248.7 nm).

A TALMS signal was obtained with p-xylene and the Si 252.8 nm line. The intensity of this signal was increased by heating the cell. TALMS signals were also found at the Co 252.9 nm, Sb 252.85 nm, and Pt 273.40, 248.72 nm lines. No TALMS signals were found for o- or m-xylene. A large benzene interference was found at the Si line. This Si line (252.85 nm) is very close to the Co line used to obtain a benzene TALMS

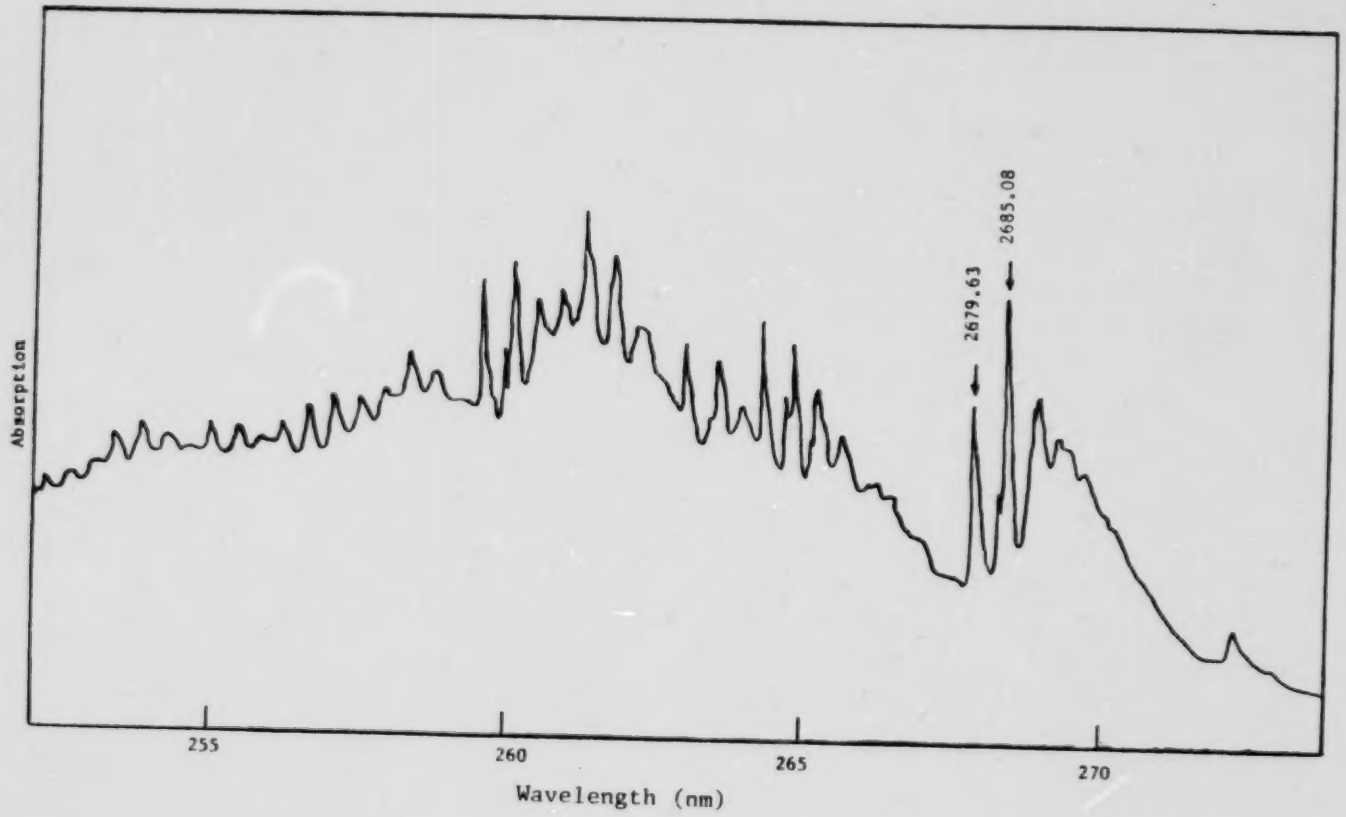


Figure 7. Medium resolution absorption spectrum of o-xylene.

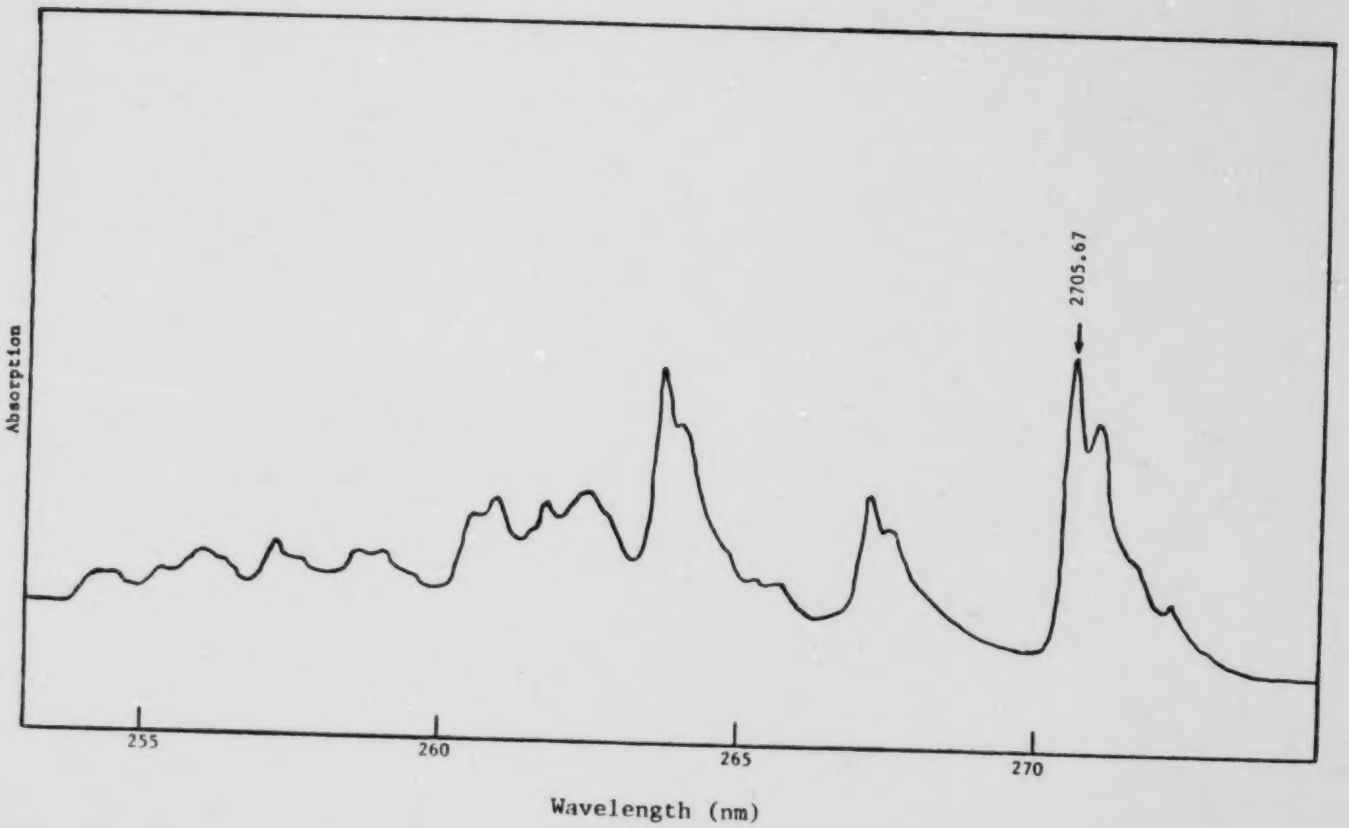


Figure 8. Medium resolution absorption spectrum of m-xylene.

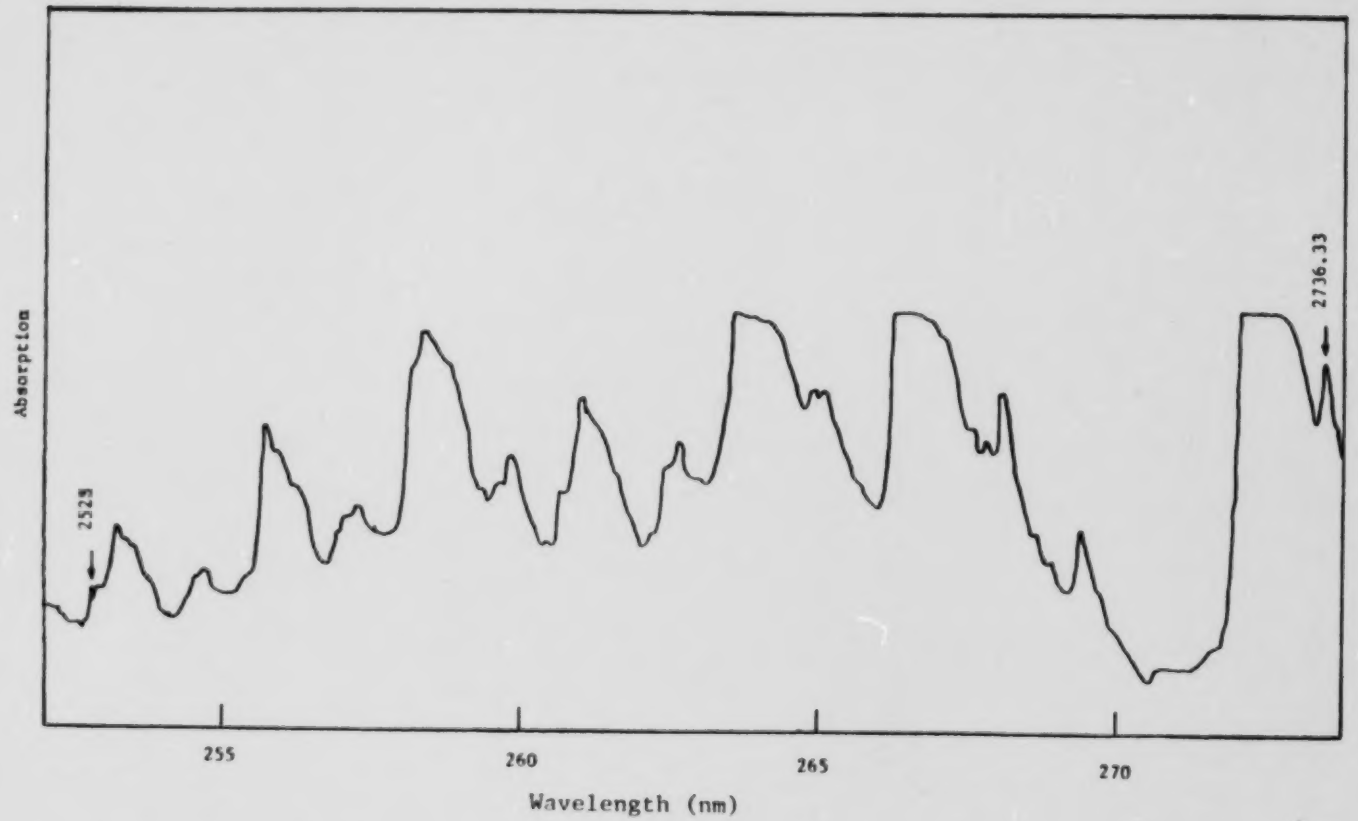


Figure 9. Medium resolution absorption spectrum of p-xylene.

signal (252.897 nm). Hence, a Co lamp was made and a TALMS signal search was made using o, m, and p-xylene. A good signal was obtained for p-xylene with the Co 252.897 nm line,but no signals were obtained for o- and m-xylene.

Three lines near 252.9 nm were found giving good TALMS signals with p-xylene and each of these lines also give benzene signals. The signals could be due to benzene contamination of the xylene samples. To resolve this question, gas chromatograms were obtained of the p-xylene used in these experiments. The results are displayed in Figures 10 and 11. Figure 10 is the record obtained when 50 uL of p-xylene vapor was injected into a gas chromatograph. Figure 11 was obtained when a mixture containing 25 uL of benzene vapor and 25 uL of p-xylene vapor was injected. Unfortunately, the minimum detection level for benzene is not known, but there is every indication that any benzene impurity in the p-xylene would be too small to explain the TALMS signals.

Bromobenzene

A TALMS signal was observed for the sharp absorption feature of bromobenzene at 266 nm using the 266.466 nm Fe II line. This match was obtained by superimposing a Fe emission spectrum on the bromobenzene absorption spectrum using a medium resolution spectrograph. The Fe line results from a transition between the $^2F_{7/2}$ and $^2G_{9/2}$ states. Hence, a complex Zeeman pattern will result. A recording of the bromobenzene absorption spectrum is presented in Figure 12.

The high resolution spectrum of bromobenzene in the 36,991 cm^{-1} (270.0 nm) region has been published by Cvitas [6]. A TALMS signal was obtained using this absorption feature and the Pt 36,993.6 cm^{-1} (270.2 nm) line. The resulting TALMS signal is displayed in Figure 13.

Aniline

A high resolution spectrum of aniline was published by J. Christoffersen et al. [7] in 1969. They report sharp absorption features in the region between 34,037.6 and 33,996.6 cm^{-1}. The Bi 34,023.17 cm^{-1} (293.8 nm) line was used to obtain a TALMS signal. Figure 14 shows the TALMS signal.

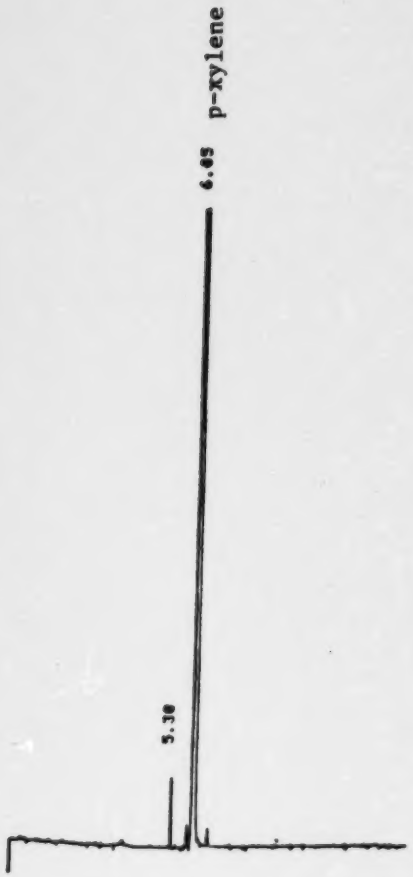


Figure 10. Chromatogram of p-xylene headspace.

Figure 11. Chromatogram of p-xylene and benzene headspace.

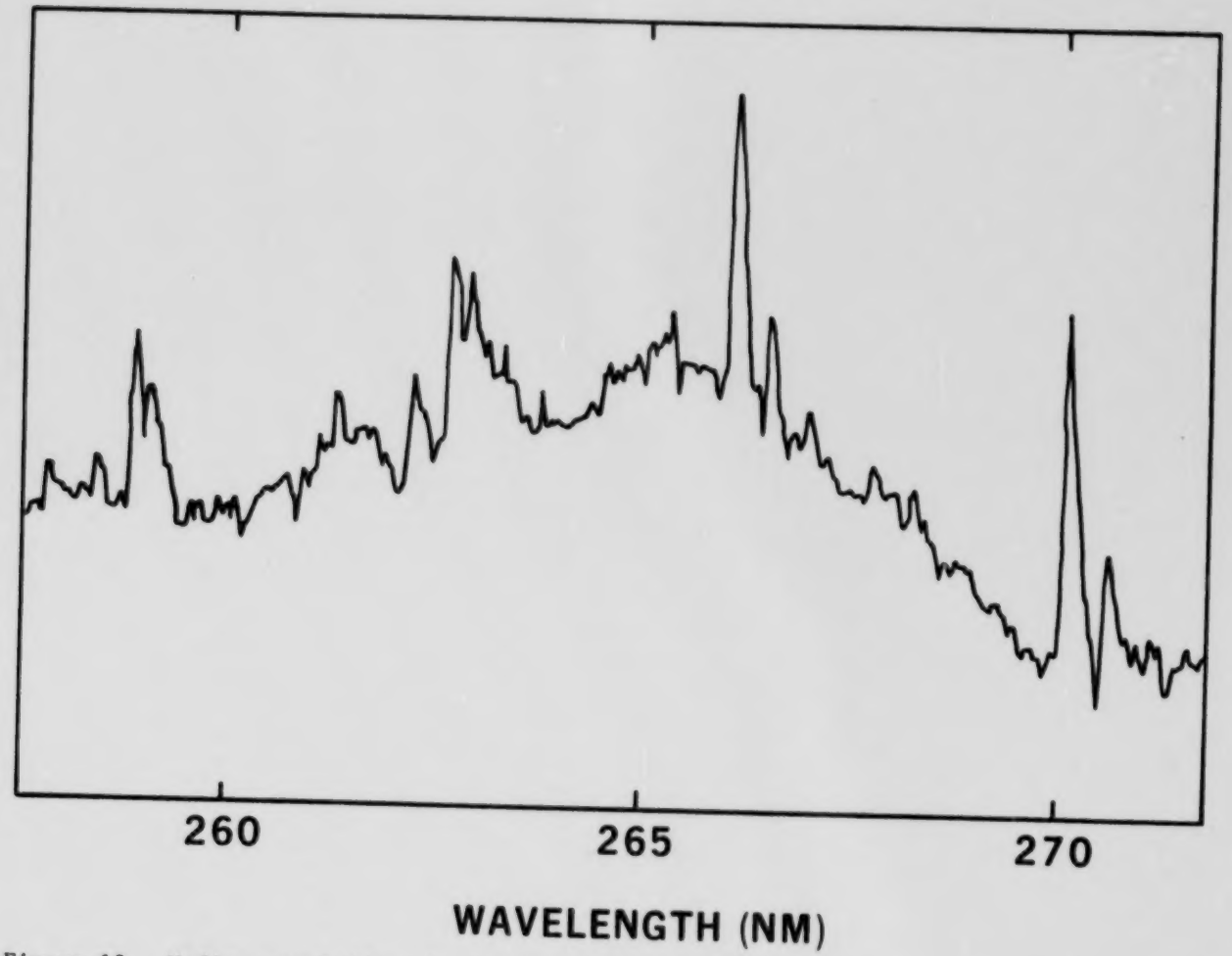


Figure 12. Medium resolution absorption spectrum of bromobenzene.

XBL 841-558

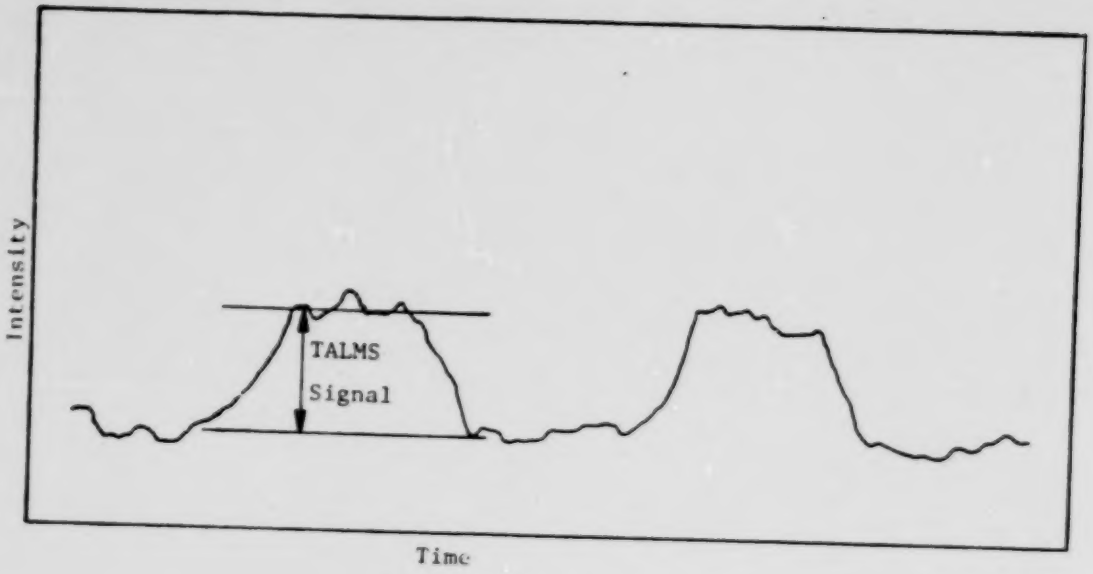


Figure 13. TALMS signal for bromobenzene using the Pt 270.2 nm line.

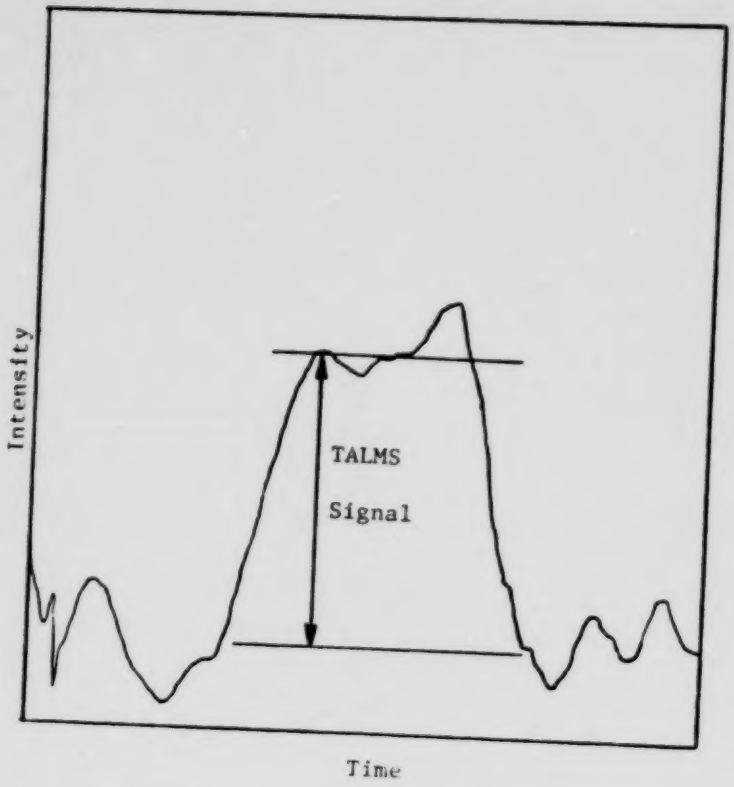


Figure 14. TALMS signal of aniline using the Bi 293.8 nm line.

Thus far TALMS signals have been found for the molecules and atomic lines listed below (in nm):

	Co	Ag	Hg	Fe	Si	Pt	Bi	Sb
Benzene	252.9		253.7		252.8			252.9
Bromobenzene				266.5		270.2		
Chlorobenzene			253.7					
Toluene			253.7					
P-Xylene	252.9				252.8	248.7		252.9
						273.4		
Aniline							293.8	
Phenol				275.0				
Pyridine			253.7					
Formaldehyde		338.3						

SECTION 5

LAMP DEVELOPMENT

Successful use of the TALMS technique depends upon the availability of stable and intense emission lamps that can be used in a strong magnetic field. The last requirement excludes the use of bulky hollow cathode lamps. Two types of lamps have been developed for tunable atomic line molecular spectroscopy (TALMS) techniques. One depends upon a magnetically contained discharge and has the potential of exciting the spectrum of any element at high intensity and low noise level. In its present form the discharge takes place in a flowing gas stream. Although it should be possible to construct a sealed off version, this has not been done as yet. The other type of lamp is sealed off, which means it is more convenient to use, but only the more volatile elements, e.g. Hg, As, Pb, etc., can be excited.

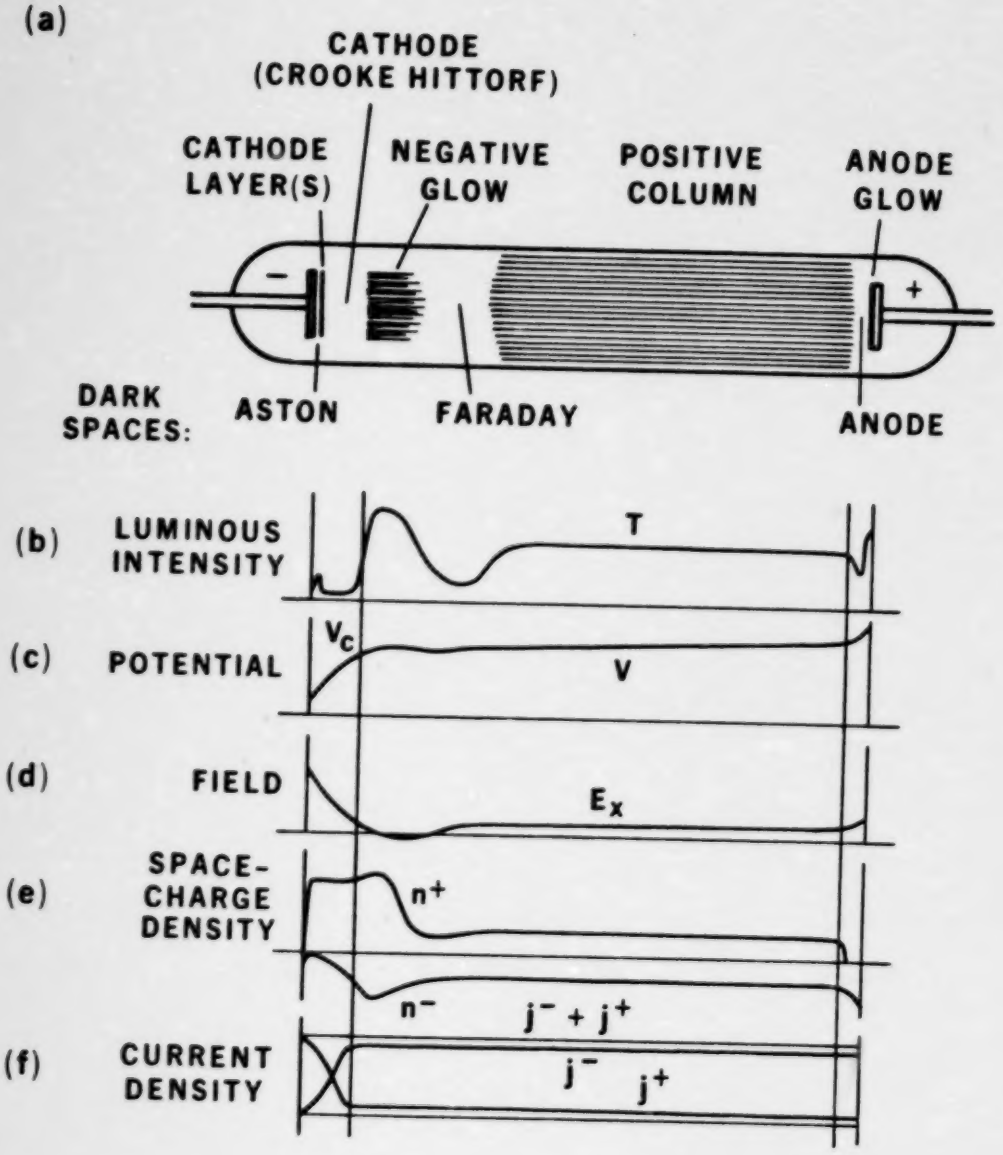
Magnetically Contained Lamp

The magnetically contained discharge lamp (MCL) has been improved so that it is stable, intense, and long-lasting and emits multi-element spectra both in parallel and perpendicular magnetic field directions. It is not possible to utilize TALMS techniques without this type of light source [3,4].

To understand the operating mechanism of the MCL, the elementary principles of light emission from electrical discharges must be considered. The first part of this description is devoted to the mechanism of light emission from a purely physical point of view. Next, the effect of a magnetic field on this discharge will be discussed since this is an integral feature of MCL operation.

The principal phenomena associated with a normal glow discharge are shown in Figure 15 [8]. In this figure:

I = intensity of emitted light
V = electric potential (volts)
E = electric field (V/m)
n^+ = density of positive charges (coulombs/m^3), mostly ions
n^- = density of negative charges (coulombs/m^3), mostly electrons
j^+ = current of positive charges (amperes/m^2)
j^- = current of negative charges (amperes/m^2)



XBL 845-1878

Figure 15. The normal glow discharge in neon in a 50 cm tube at 1 torr pressure. The emission regions are shaded.

Light from a conventional hollow cathode lamp (HCL), as well as the MCL, originates at the negative glow region; whereas light from a neon sign originates in the positive glow region. Light from an electrodeless discharge lamp also originates from the positive column. We shall concentrate on the cathode dark space and the negative glow region since both regions are tightly coupled in the process of light emission from the MCL.

In Figure 15c notice that the electric field potential does not vary in a linear fashion between the cathode and the anode. This is due to the uneven distribution of space charge in this region. The potential drop in the discharge tube can be divided into three regions:

V_c = voltage drop in the region of the cathode

V_p = voltage drop in the positive column

V_a = voltage drop in the region of the anode

(For this discussion the voltage drop between the onset of the positive glow and the negative glow is included as part of the positive column potential drop.) The total voltage across the discharge tube is then:

$$V_t = V_c + V_p + V_a$$

The anode potential is just the ionization potential of the gas in the discharge tube (ca. 10 volts), and the potential drop in the positive column is generally small (a few V/cm). The voltage drop in the region of the cathode is on the order of 100 volts. Hence:

$$V_t = V_c$$

To understand the nature of the light emission from this region, remember that a) the electric field near the cathode is a linear function of distance, and b) most electrons are emitted from the cathode because of ion bombardment. Very close to the cathode (the Aston dark space) the electrons have not accelerated through a potential drop sufficient to impart the energy required for atomic excitation or ionization. In addition, the ions moving towards the electrode have a velocity such that combination with electrons in the vicinity (with subsequent emission) is unlikely. Also, excited state lifetimes are such that ions that were in excited states after formation have already undergone radiative decay to their ionic ground states. Hence, no light is emitted from this region. The cathode layer appears at the point where the few ions that have been excited return to the ground state, but this is a small effect. In the cathode dark space, electrons have been sufficiently accelerated by the electric field to cause ionization. Secondary electrons result, but their energy is too low to cause atomic excitation, while the energy of the primary electrons that caused them is still too great. Also, the probability of collisions with primary electrons that result in atomic excitation is small. Hence, there is little light emitted from this region.

In the negative glow region a large number of secondary electrons begin to excite gas atoms. The density of ions is great (Figure 15e), and there are many radiative recombinations. This is the reason for light emission

from the HCL and MCL. Early in the development of the MCL it was observed that cathode erosion occurred in an unusual manner. Instead of random wearing away of the electrode, a sharp, line-like crevice appeared after running for extended periods of time. It is now realized that this results because the magnetic field concentrates the positive ions into a plane perpendicular to the field direction. This has the effect of increasing the intensity of emission lines from an MCL over that from an HCL.

Initially, lamps were constructed with a graphite cup for the cathode. This cup is filled with the desired material, and the spectrum obtained with a pulsed (5 - 50 kHz) DC discharge. The surrounding pole pieces and housing of the lamp (see Figure 16) are grounded, and the cup cathode is charged to -500 v. The lamp is oriented for use in the direction perpendicular to the magnetic field. As the discharge continues, a slit is cut into the cup as indicated in Figure 17A. This arrangement produced a stable discharge, but after a few hours the discharge region was located inside the graphite cup such that the volume of highest intensity could no longer be focused on the spectrograph slit (Figure 17B). The sputtering process begins at the center of the cup but works its way over to one side and behind the graphite wall as it continues.

It was found that the intense discharge region could be held in view for 70 hours by surrounding the graphite cup with a boron nitride insulator that had a slit cut in one edge (Figure 18). Again a slit was cut into the graphite cup by the discharge, but now the intense region was always visible.

Experiments were conducted to obtain an explanation for these observations. Chromium was chosen as the material to be used in these experiments because it is easy to sputter but requires high power and high temperatures. These characteristics simplify observation of the sputtered region and, at the same time, test the operation of the equipment at high levels of input power. In the first experiment a cathode cup was constructed without the graphite by pressing Cr powder into a boron nitride insulator that had a slit cut in the top (Figure 19). This arrangement produced stable intense Cr spectra for ca. 15 hours, but then the arc begin to wander over the face of the boron nitride. Upon opening the lamp housing, it was found that a layer of Cr had coated the region outside the slit. This conducting layer was responsible for the arc wander. Before formation of the coating the sharp edges of the slit produced an electric field gradient that focused the Ar^+ ions into the region containing the packed Cr powder. Since boron nitride is an insulator, no sputtering occurred on its surface until it became coated with a conducting film. The presence of the film would not have altered the discharge characteristics of the graphite cup in earlier experiments since it is already a conductor.

Aluminum has always been an element whose spectrum is difficult to excite in a stable manner. This is probably related to the ease of formation of an oxide coating on this material. A cathode was made using the arrangement in shown Figure 18 with Al powder filling the graphite cup. Although an Al spectrum was obtained, the discharge was unstable with the occasional emission of bright flashes. No improvement occurred when a solid Al rod was substituted for the powder. The surface of the material looked blackened after the runs as though oxidation had taken place.

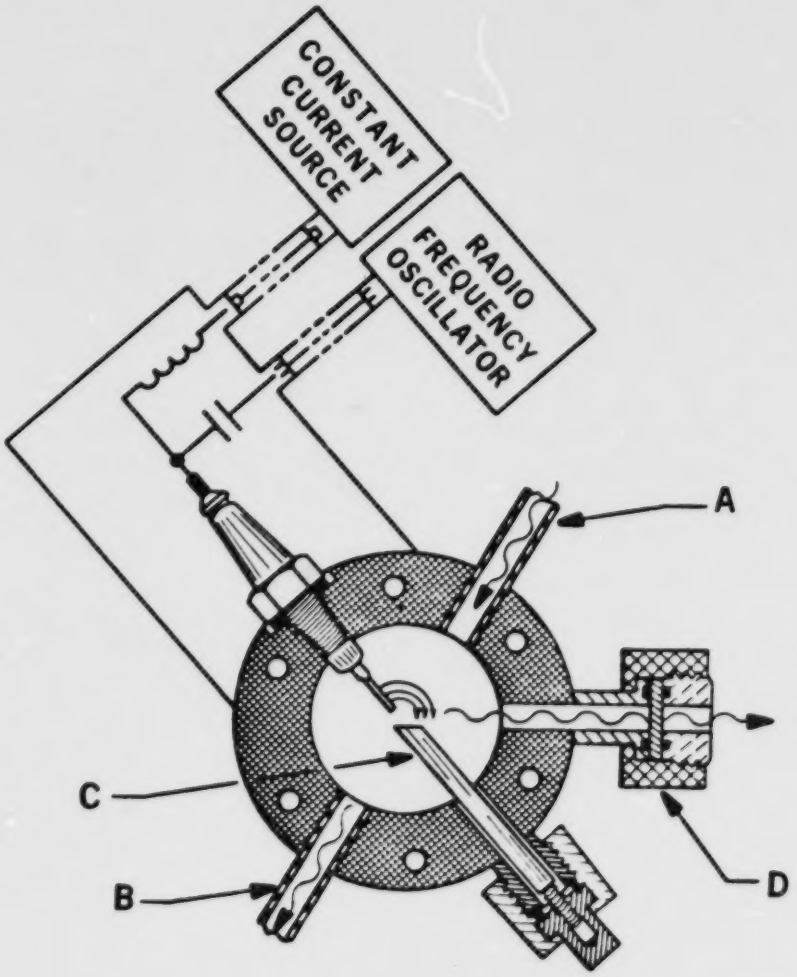


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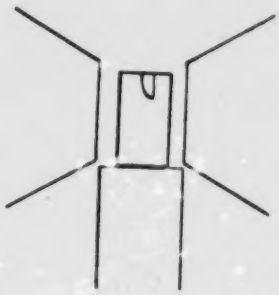
Figure 16. Magnetically contained discharge lamp. The carrier gas enters port A and exits port B; C is the metal cathode and D is the window assembly.

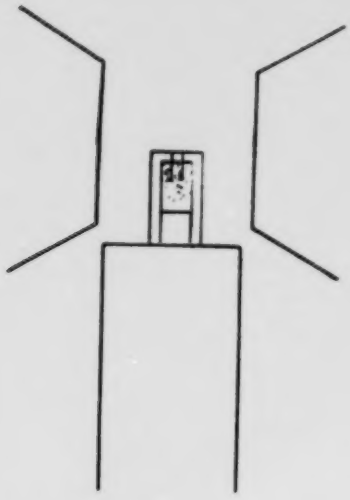


A

B

Figure 17. Pattern cut in a graphite cup by a magnetically contained discharge. A: View along the optic axis. B: Overhead view showing the region of highest intensity.

Figure 18. Graphite cup with a boron nitride outer shell.



A

B

Figure 19. Boron nitride cup. A: Cutaway view along the optic axis; B: View from the top.

The addition of 5% hydrogen to the flowing gas stream improved this situation. A steady discharge was maintained for 20 hours, and the surface of the metal was shiny after these runs both with the Al metal plug and the Al powder. Next a 50:50 mixture of Al powder and graphite was tried without hydrogen in the gas stream. This also produced a stable long-lasting discharge and was felt to be preferable since it avoids the need to work with hydrogen.

In order to optimize the graphite cup - boron nitride shell configuration, an arrangement was investigated with the shell extending several mm above the cup (Figure 20). This caused the sputtering Ar^+ ions to strike the material only through the slit rather than both through the slit and over the top as had occurred before. With the boron nitride shell either above or level with the graphite cup, stable, long-lasting discharges of Cr were obtained. It is not clear that one is to be preferred over the other. However, there may be important differences when volatile elements (e.g. As, Se, etc.) are to be excited.

The slit is cut into the graphite cup because of a focusing effect of the sputtering atoms (Ar^+) by the magnetic field. It was found that by placing the cathode three to four mm under the center of the magnet pole pieces a small defocusing effect appeared that makes the sputtered area larger. This configuration led to longer lifetimes for Al and Cu lamps. In an effort to understand this focusing effect, a series of experiments were carried out with a solid Cu cathode with no boron nitride shell or graphite cup. The effect of various magnetic field shapes on the pattern cut into the cathode was studied. Different magnetic field gradients were produced with pole pieces of the shapes sketched in Figure 21. The slit cutting action was only observed with the arrangement sketched in Figure 21c. This is consistent with the calculated motion of an Ar^+ ion in a 500 volt electric field and a 10 kG (1 T) magnetic field. The equations governing this situation were developed to describe the motion of ions in a cyclotron accelerator. As described by D. L. Cohen [9], a magnetic field of the shape in Figure 21c will cause Ar^+ ions to move in circles of two mm radius and be focused into a median plane; whereas the field shapes in Figures 21a and 21b do not result in focusing action. This calculation also predicts that if the magnetic field direction is reversed, the cutting (focusing) will occur on the opposite side of the cathode. This effect was verified by rotating the lamp housing 180 degrees.

Magnetic field focusing causes the ion beam cross section to be smallest at the center of the field. As the beam cuts deeper into the cup, it travels past the center of the field before striking the material to be sputtered. This is responsible for the defocusing effect. The shape of the induced electric field around the boron nitride shell reinforces the magnetic focusing when the sputtered material is high in the cup. This effect no longer operates as the beam cuts further into the cup.

The cutting pattern that results when the lamp is arranged for viewing parallel to the magnetic field direction is also consistent with this calculation (see Figure 22). In this case the Ar^+ ions cut a circular pattern into the material being sputtered. When the hole in the boron nitride shell and graphite cup was two mm in diameter, a four mm diameter ring was produced in the region of the sputtered material. When the hole was one mm in

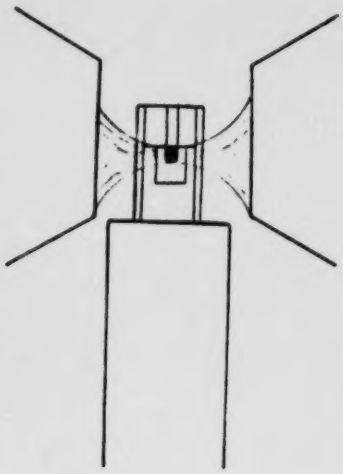


Figure 20. Discharge pattern with high boron nitride shell. (Location of interior graphite cup is indicated.)

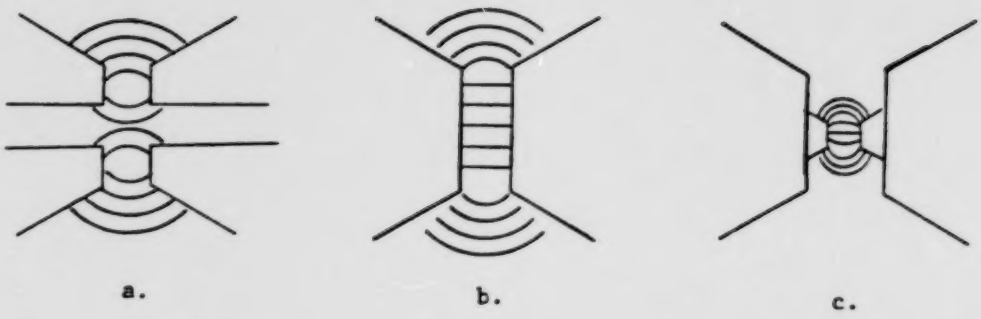


Figure 21. Field shapes used in the investigation of the magnetically contained lamp.

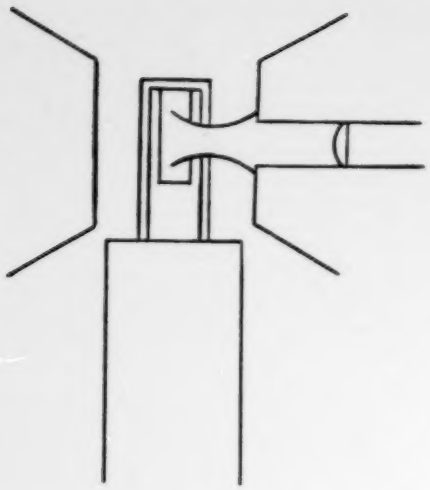


Figure 22. Shape of the discharge with the parallel Zeeman configuration.

diameter, no circular pattern resulted. This is consistent with the ion motion discussed earlier.

Some theoretical work was done to understand the mechanism of the MCL discharge. We discovered that the discharge is confined due to the focusing effect of the ions, that it is characterized electrically by an abnormal glow, and the light emitting process is due to an arc discharge [8]. The theory explains the stability and very high light emission that results from this configuration.

Finally, an arrangement was perfected that eliminated the graphite cup by providing a conducting path to the material to be excited contained in a boron nitride cup. This extended lamp life times to ca. 200 hours. Thus far, lamps of the following elements have been made:

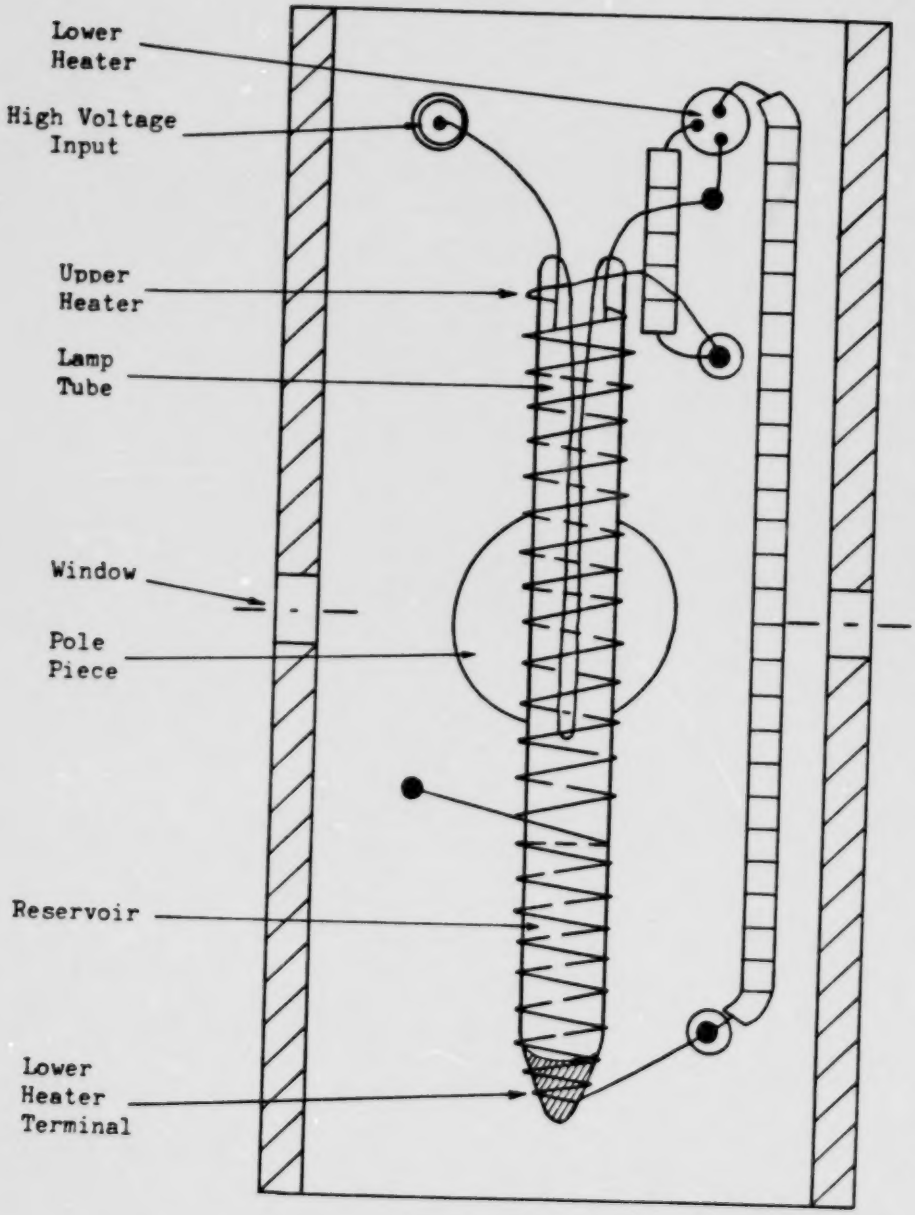
Fe	Cr	Co	V	Al (graphite)
Rh	Cd	Zn	As	Ag
Cu	Ge	Au	Se	Hg
Bi	Sb	Pt	Mo	Ga
Mn	Si	Pb	Ir	Ni

The magnetically confined lamp has now been developed to a point where commercial production is feasible. The customer can be supplied with cathodes of a variety of materials whose operating characteristics have been determined. At the present stage of development these cathodes will be capable of many hours of operation at high power levels.

Low Frequency Discharge Lamp

In addition to the MCL, a sealed off lamp system that operates continuously for about one year has been developed for various elements. The main difference between the MCL and the low frequency discharge (LFD) lamp is that the latter operates in a fashion similar to an electrodeless discharge lamp (EDL). The LFD emits very intense resonance lines but a smaller number of lines than other types of discharges. The advantages of the LFD over the MCL are: extremely long life, ease of use, and intense resonance lines. Figure 23 illustrates the construction of the LFD.

Power is coupled into the lamp through two electrodes located in either end of a U shaped tube. The lamp is contained in an aluminum housing. In the center of this housing are two iron pole pieces that contact the yoke of a permanent magnet when it is installed for use. Heating coils wrapped around the lamp allow control of the temperature and hence the vapor pressure of the element in the lamp. The bottom portion of the U tube contains the element whose spectrum is to be excited and also serves as a reservoir for the carrier gas that maintains the discharge. The upper coil is used to make sure the element does not condense in the upper regions and block out the emitted light. These lamps are commercially available from W. C. Heraeus GmbH, PEW, Hanau, West Germany.



XBL 8010-12658

Figure 23. Low frequency discharge lamp.

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APPENDIX A

INSTRUCTION MANUAL
FOR
TRANSPORTABLE TALMS

Start Up Procedure

1. Switch both photomultiplier tube (PMT) power supplies to manual. Turn on the instrument using the switch on the power supply. Allow one hour warm up time.

2. Set the compensation plate perpendicular to the light path.

3. Check the output from each PMT power supply. These should be set to cause a displacement on the oscilloscope of between 0.5 and 0.6 volts.

4. Switch both PMTs to automatic gain control (AGC). Set the AGC level to between 0.5 - 0.6 volts. Although the exact value is not important, both PMTs should be, as nearly as possible, at the same AGC level. This can best be achieved by looking at the PMT difference output. It should read 0 volts on the DC voltage setting.

5. If the shape of the sinusoidal PMT output is disorted on the oscilloscope, adjust the squeezer frequency, amplitude, and DC level to correct it.

6. Tune the lock-in amplifier (LIA) as follows:

   a. Switch the AGC difference output to Log 2 "off". This causes the input to the LIA to be the AC signal from PMT 1.
   b. Set the gain to about 2.0
   c. Hold in the red button and tune the LIA by turning the frequency knob until the AC signal on the scope is maximized. Be sure that the frequency is the same as that for the squeezer and not a higher harmonic. The squeezer frequency can be monitored using the output on the squeezer panel. The button will return to the phase adjust position when released.

7. To adjust the phase, release the red button. This causes the mixer signal to be displayed. Make the display as symmetrical as possible by adjusting the phase knob.

8. Switch the AGC back to the Log 2 - Log 1 position.

9. Adjust the compensation plate so there is no AC signal. This compensates for self-absorption in the lamp.

10. Now, look at the DC output. Set the time constant to one second. Adjust the DC offset so that the output is zero with no benzene in the cell.

11. Change to a time constant of 30 seconds. Adjust the LIA gain to whatever is appropriate for the level of noise associated with the DC output of the LIA.

Optical Components

1. The light source is a mercury Pen Ray Lamp, 6.5 mm diameter, mounted in an aluminum housing. This housing is mounted in a permanent magnet that is secured to the optical bench.

2. Knobs on the lamp power supply allow for amplitude and frequency adjustment. The presence of standing waves of plasma in the lamp indicate poor power coupling and unstable operation. Alter the amplitude and/or frequency until standing waves disappear.

3. The Pen Ray Mercury Lamp used in this instrument excites a discharge in a U tube. This tube should be oriented so that an imaginary line connecting the legs of the U (∪) is parallel to the optic axis.

4. The lens has a focal length of 70 mm and is constructed of ultraviolet transmitting quartz.

5. The compensation plate is constructed of UV grade fused silica and can be rotated to make an angle between 0 and 90 degrees with the optical axis. A wing nut allows the plate to be clamped into position at the desired angle.

6. The squeezer is a quartz slab that can be subjected to varying amounts of pressure by a surrounding electromagnet. The power supply for this magnet allows both constant pressure and pulsed pressure to be applied.

7. The beam splitter is a half silvered mirror that can be rotated to make an angle between 0 and 90 degrees with the optic axis. A linear polarizer and a 253.7 nm interference filter are mounted at the entrance of the beam splitter housing. The reference PMT is also mounted on this housing. Both the reference and signal PMTs are type 1P28.

8. The sample cell is six inches long with entrance and exit ports at either end. The windows are held on the cell with stainless steel flanges that put pressure on "O-rings". These flanges should be tightened finger tight so they will not rotate. The cell is connected to a valve mounted on the side panel of the optical bench. It should be leak tested before use.

Electronic Components

1. The Eurorack contains the following components:

   lock-in-amplifier, PMT control with AGC card, lamp driver, power amplifier for the squeezer, squeezer driver, log difference card, and the power amplifier for the light driver.

2. The PMT output buffers are located on the PMT housings in the small blue Pomona boxes.

3. The lamp transformer is located on a side panel of the optical bench unit.

4. The squeezer isolation capacitors and choke are also mounted on a side panel of the optical bench unit.

5. A six pin connector supplies the units described in 3 and 4 (above) with $\pm$ 30 volts and $\pm$ 15 volts from the Eurorack. The voltages on the connectors are:

   a. +15 c. +30 e. not connected
   b. -15 d. -30 f. ground.

6. A 26 pin connector supplies power to units on the optical bench from the Eurorack.

APPENDIX B

ELECTRONIC SCHEMATICS

Lock-In Amplifier

The lock in amplifier (LIA) consists of three electronic boards: the signal board, the reference board, and a third board that provides connections to the various knobs and buttons on the front panel (the knob board). Five schematics for the LIA are contained in Figures B1, B2, B3, B4, B5. Figures B1 and B2 are circuit diagrams for the signal and reference processing electronics. Figures B3 and B4 are layout diagrams for these circuits that display the physical location of the electronic components. In Figure B5 the location and labels of the connecting pins are indicated. The pin connections between the signal board and the reference board are shown in the diagrams. The connections between these two boards and the knob board are not shown but can be easily located visually when the board is pulled out of the Eurorack.

One misleading aspect of the reference board diagram should be noted. The 10 µf and 30 µf capacitors that determine the time constant (IC10 on the reference board diagram) are actually located on the knob board and not on the reference board.

Automatic Gain Control

The automatic gain control (AGC) circuit is designed to maintain a constant DC output from the PMTs. The feedback circuit is designed so that more high voltage power is supplied when the PMT output starts to decrease and less power is supplied when the output starts to increase. The AC component at the squeezer frequency is not affected by these changes in power levels.

Figure B6 is a diagram of the electronics used to provide automatic gain control for the two PMTs. It includes diagrams of the power supply controls for the two PMTs. The ICs used are LM747s which are dual operational amplifiers. The (A) and (B) in the diagram refer to each of the operational amplifiers in the LM747s. The manual PMT power control is also shown.

Power Amplifiers

There is one schematic (Figure B7) for the two power amplifier boards. One power amplifier is required for the lamp and another for the squeezer. Both circuits are identical, and the components for each are contained on separate boards.

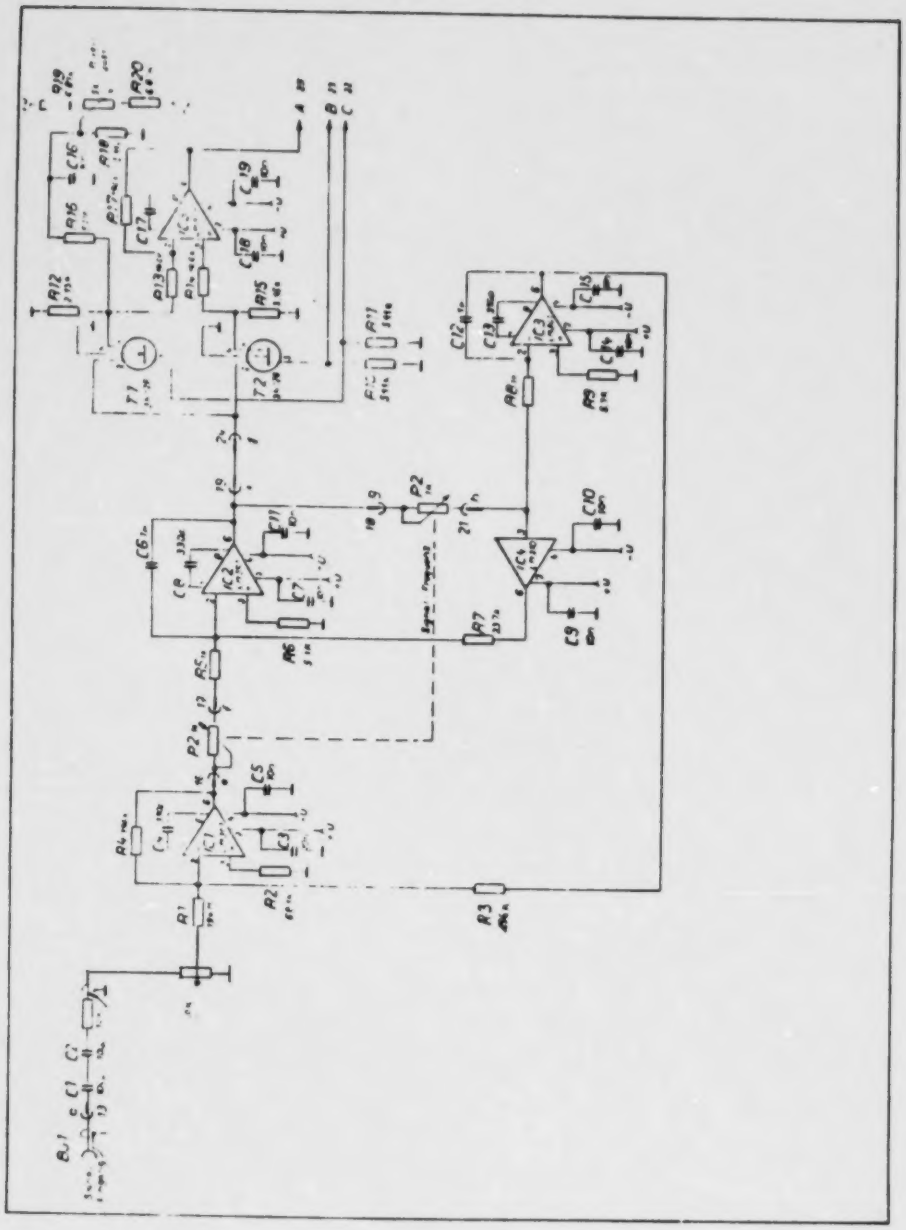

Figure B1. Lock-in -amplifier signal board.

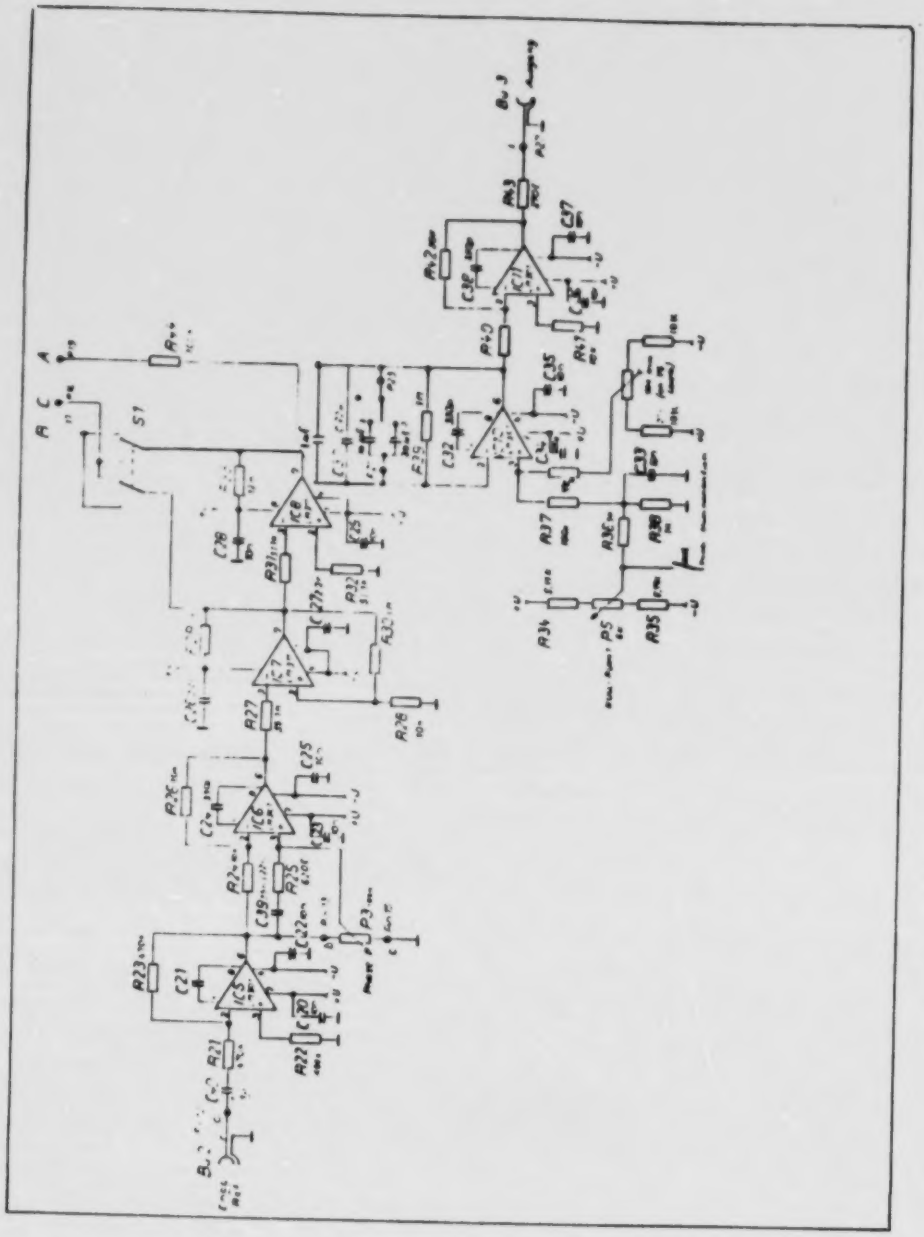

Figure B2. Lock-in-amplifier reference board.

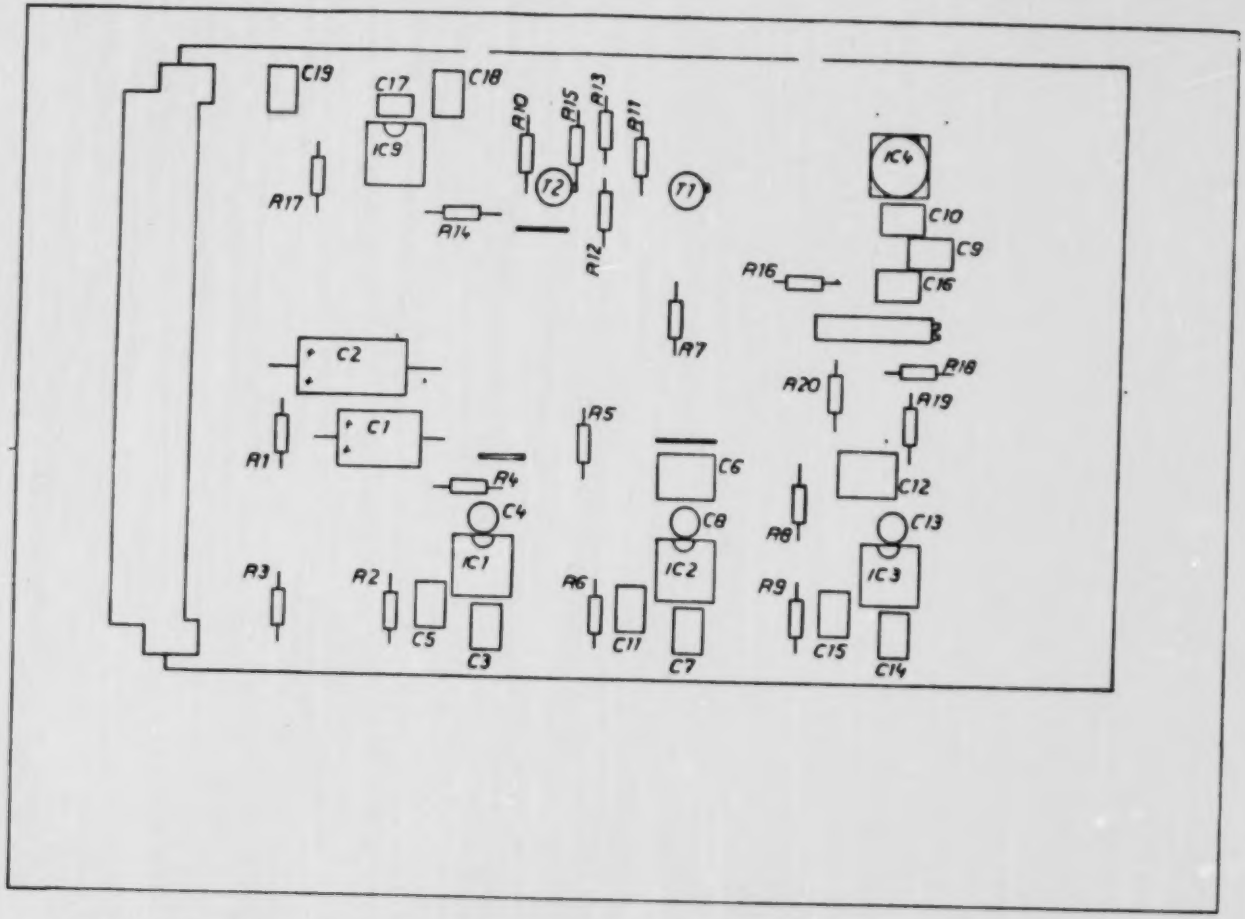

Figure B3. Component layout for lock-in-amplifier signal board.

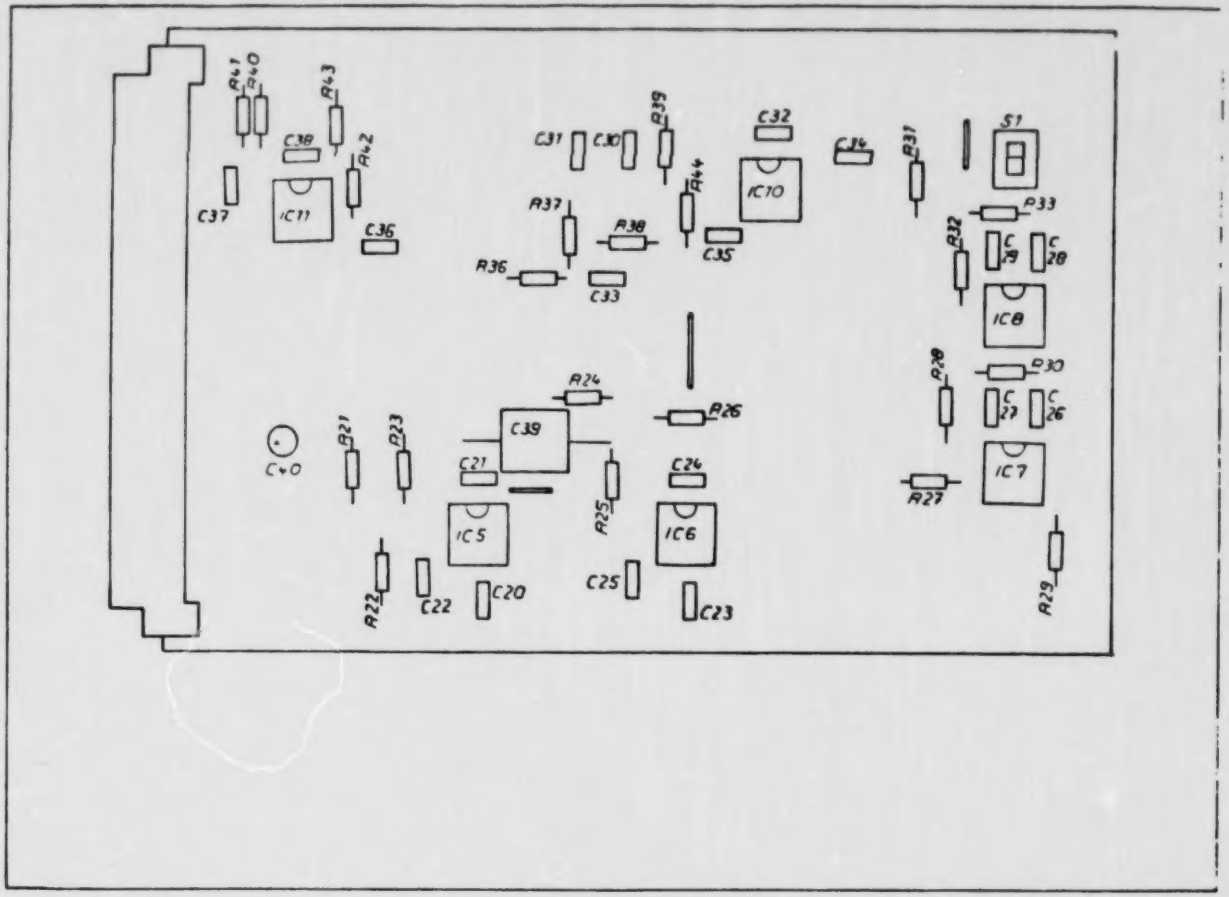

Figure B4. Component layout for lock-in-amplifier reference board.

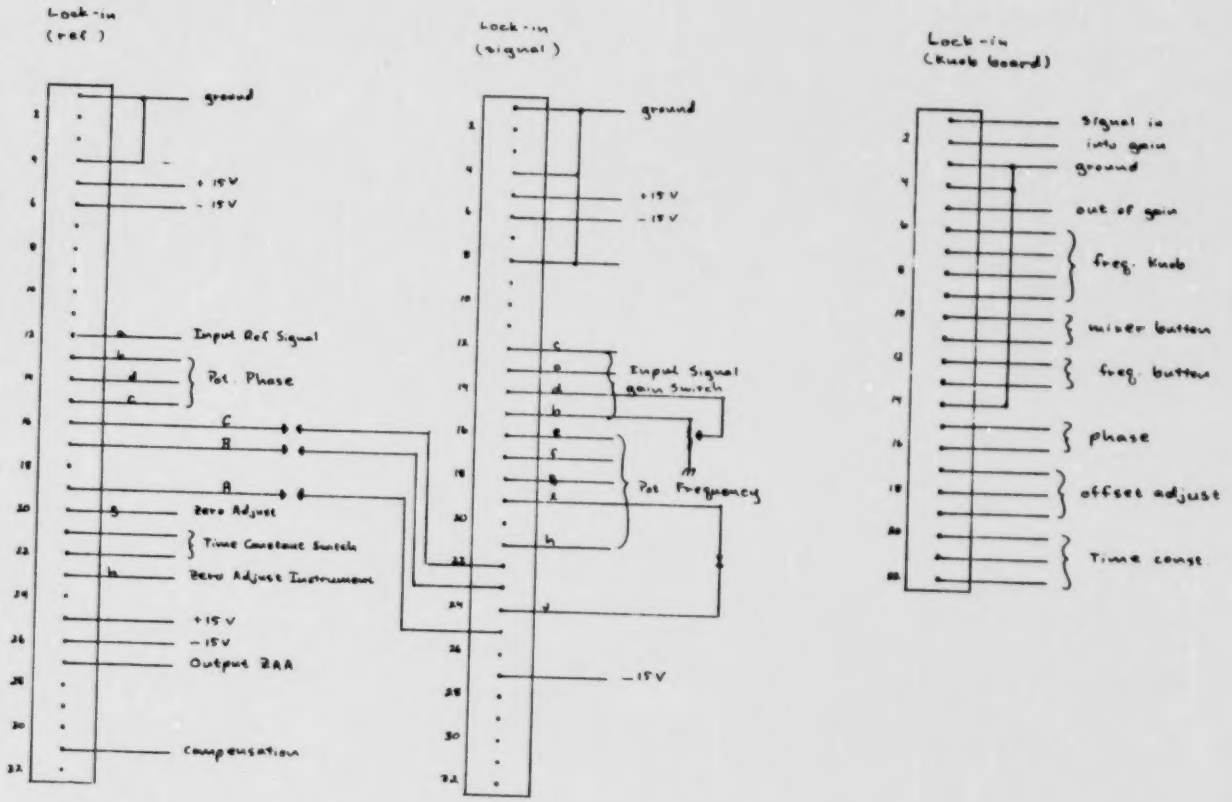

Figure B5. Lock-in-amplifier pin connections.

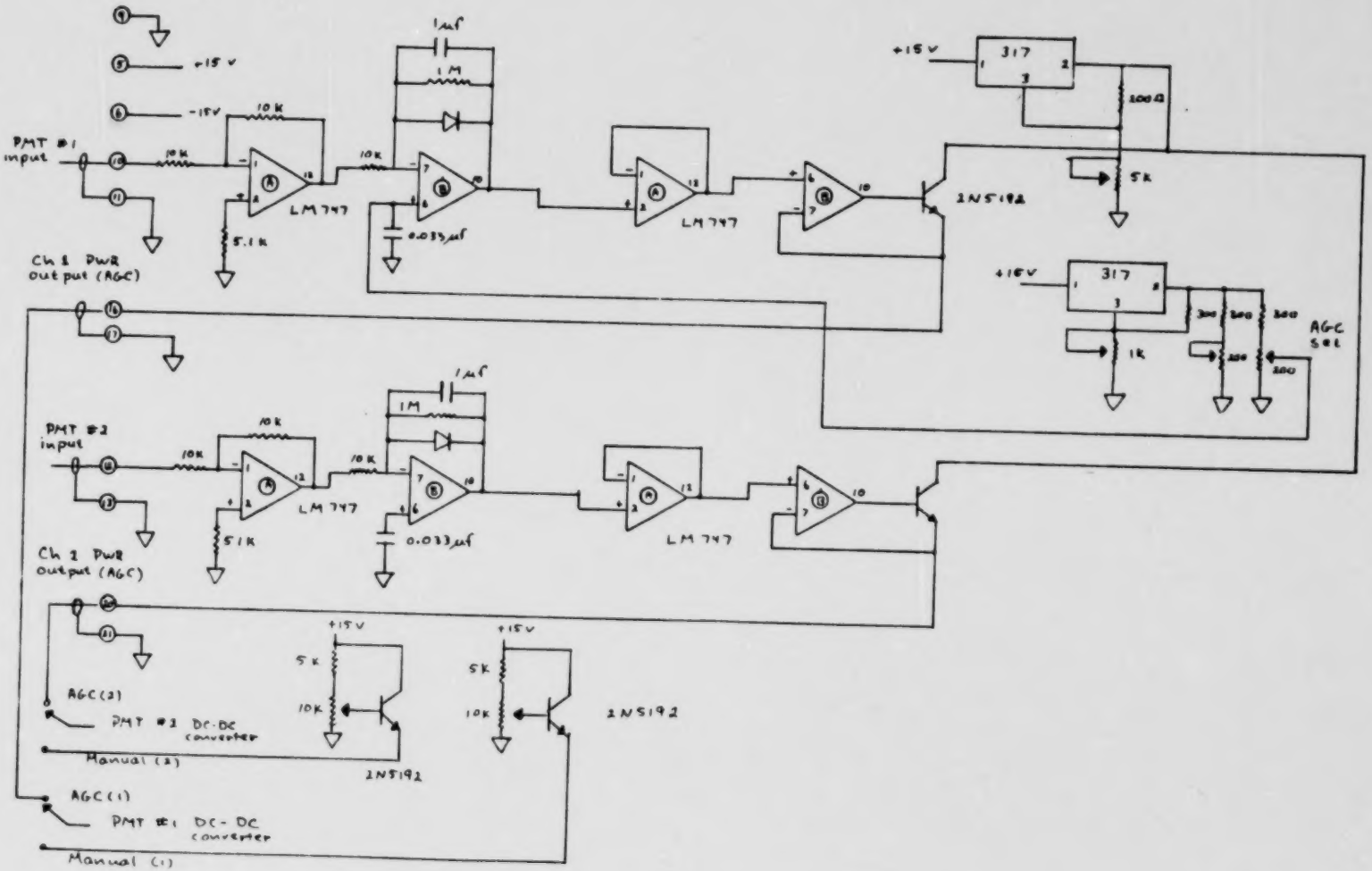

Figure B6. Two channel automatic gain control board.

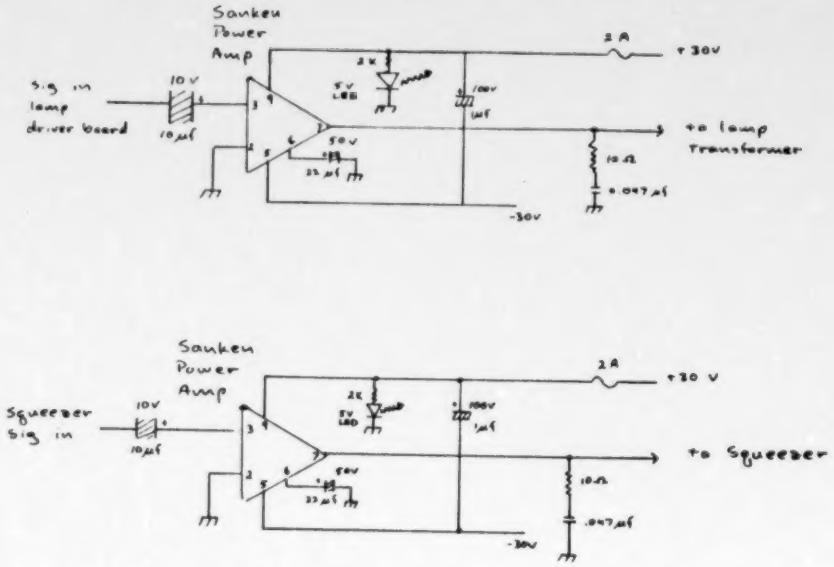

Figure B7. Power amplifier boards for lamp and squeezer.

Lamp Driver

Figure B8 is a diagram of the lamp driver electronics. The AC output from this board goes to the lamp power amplifier. This circuit contains an amplitude, frequency, and offset adjust. The location of each component is indicated in Figure B9.

PMT Output Buffer

The PMT output buffer (see Figure B10) is located in a blue Pomona box attached to the PMT housing. The PMT buffer must be located close to the PMT to prevent phase shifts in the AC squeezer frequency signal. The 100 pf capacitor at pin three of the AD503 operational amplifier is to reduce the intensity of high frequency signal from the lamp that leaks into the circuit. This capacitor can be changed to more completely reject high frequency components but care should be taken not to decrease the signal at the squeezer frequency.

Squeezer Control

A separate board contains the electronic components used for the AC and DC control of the squeezer (see Figure B11). The amplitude and frequency of the AC power may be varied, and the amount of DC power may be varied with this arrangement. A component layout is pictured in Figure B12.

Difference Log Amplifier

The circuit for logarithmic processing of signals from the two PMTs is shown in Figure B13. When the input from PMT #2 is switched off, a DC signal of one volt is switched in to prevent the AD 757N output from saturating.

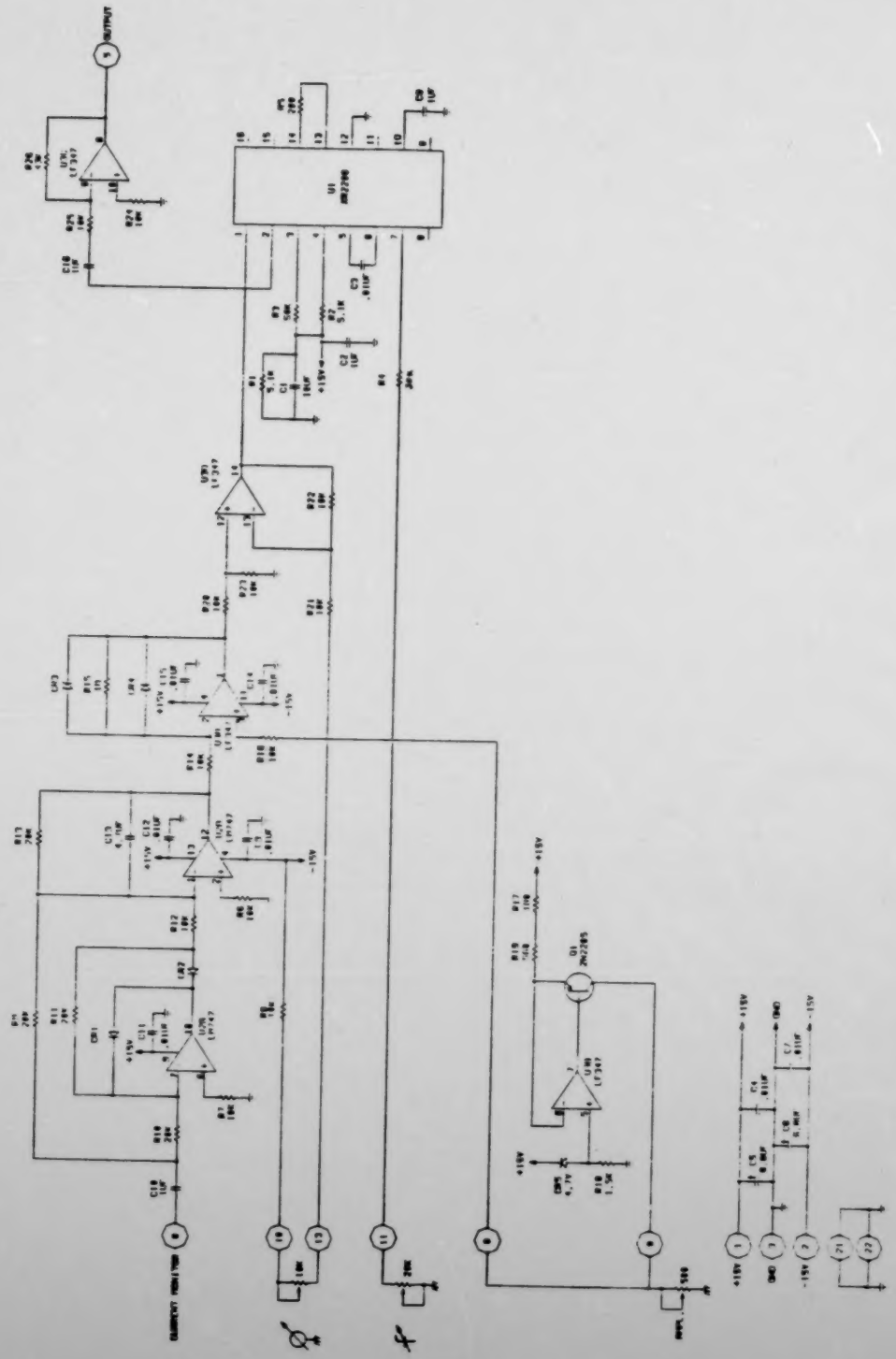

Figure B8. Lamp driver circuit.

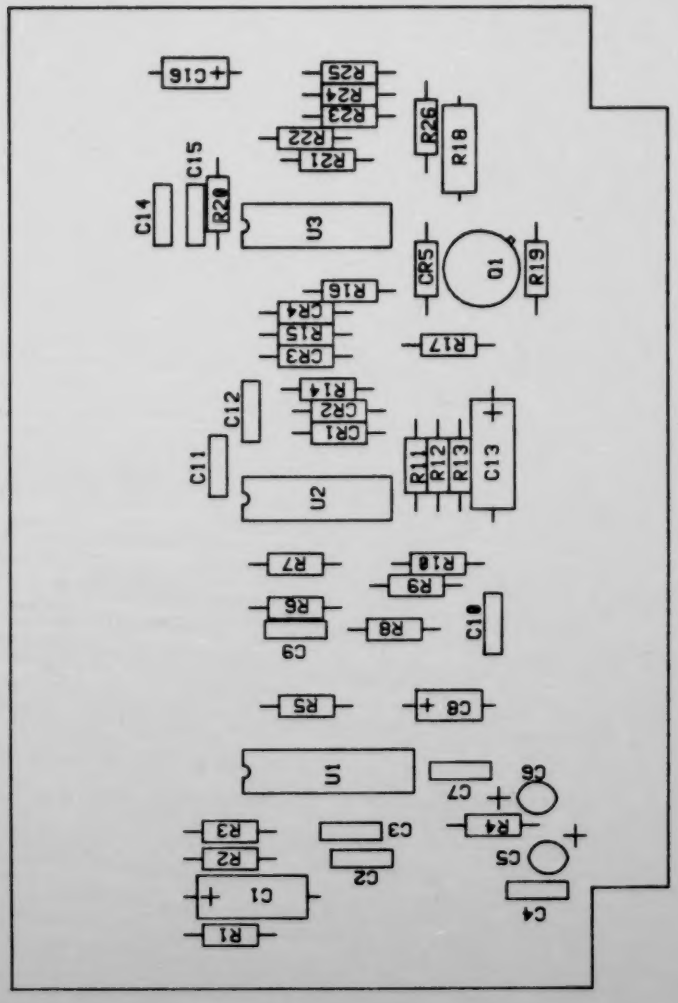

Figure B9. Component layout for lamp driver.

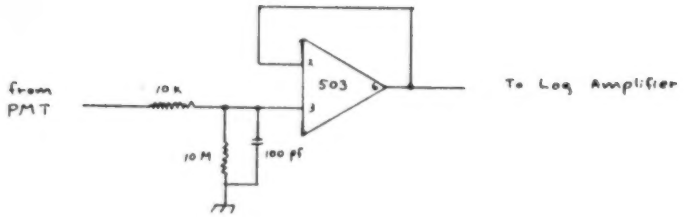

Figure B10. Photomultiplier tube output buffer.

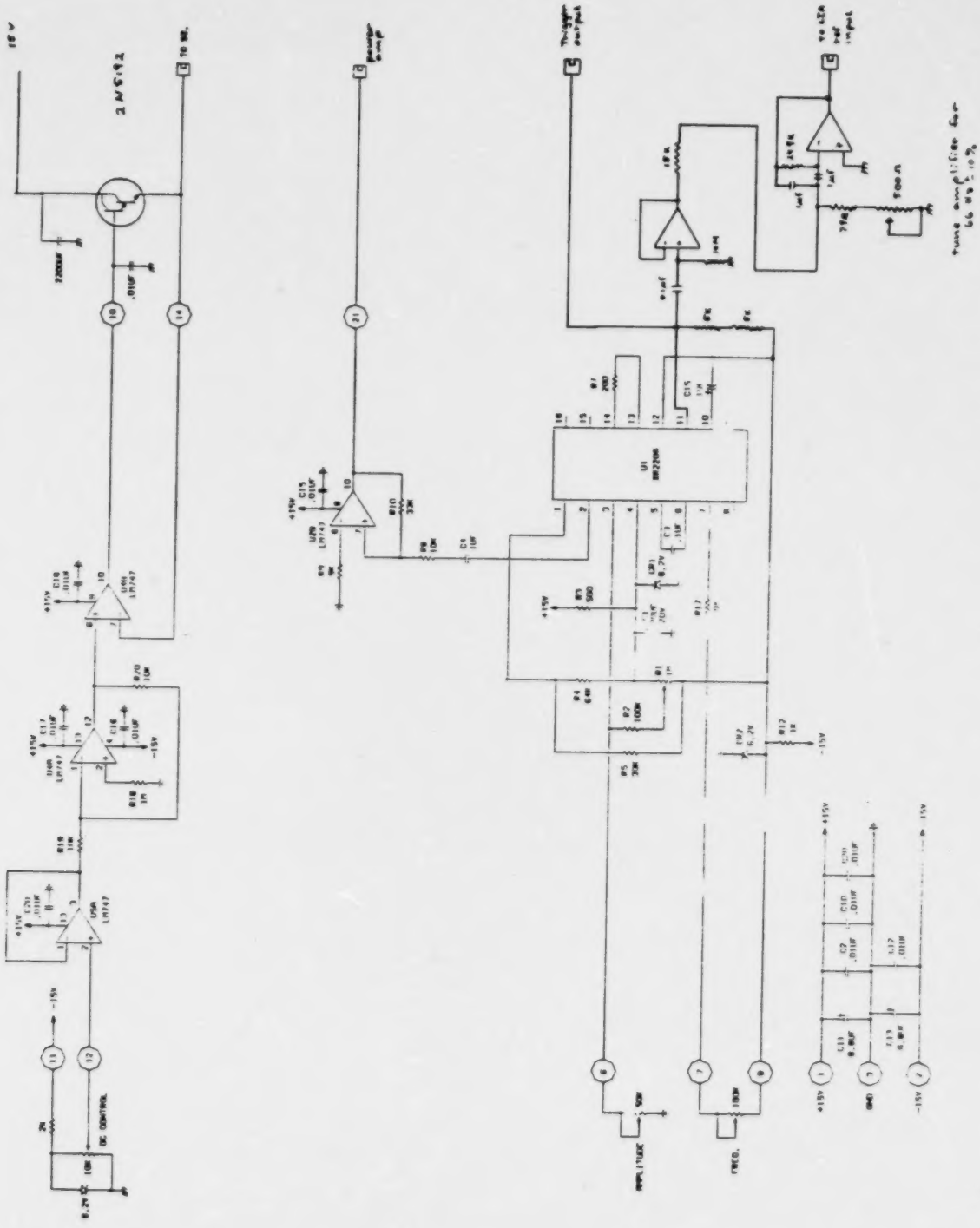

Figure B11. Squeezer control circuit.

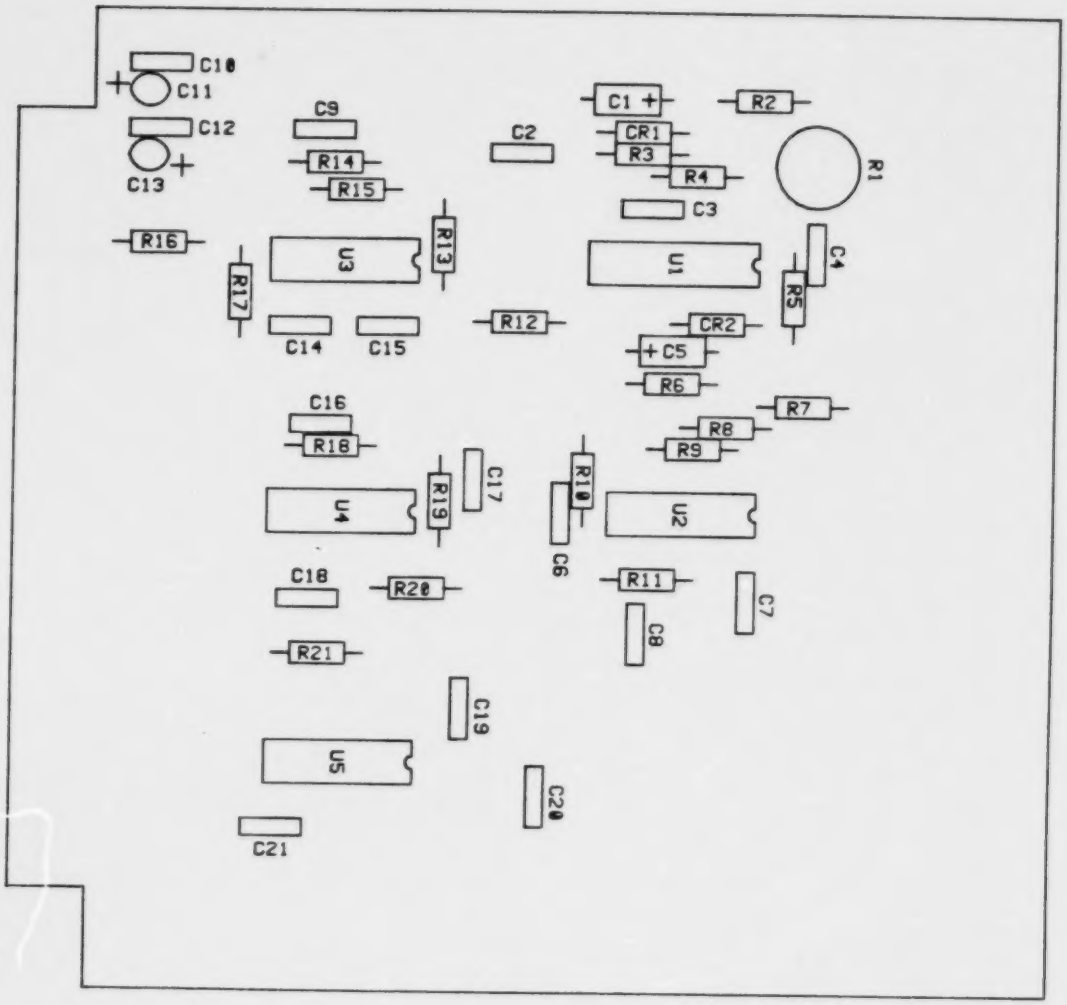

Figure B12. Component layout for squeezer.

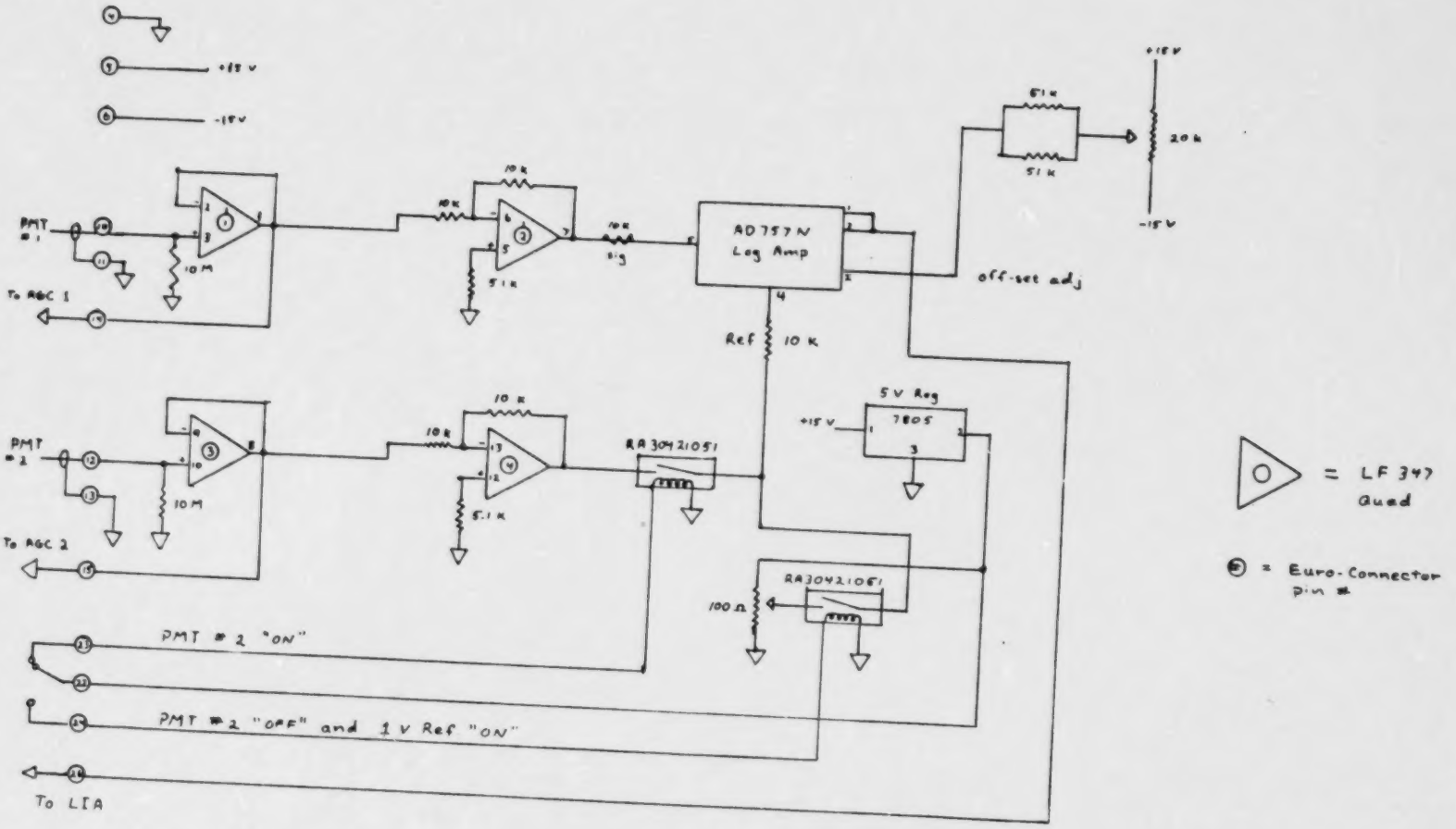

Figure B13. Difference log amplifier board.